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#### TRANSMITTAL LETTER

Sent by David Banton

Per David Banton/ah

To Environmental Protection Agency 1200 Sixth Avenue, Mail Stop HW-113 Seattle, Washington 98101 Date September 27, 1991 Project No. 913-1101

ATTENTION: Mr. Tim Brincefield

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Quantity	Item	Description		
1	copy	QA/QC Plans from Krueger Lab, Chen Northern, and Accu-Lab		
Remarks				

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## LABORATORY PROCEDURES USED IN RADIOCARBON DATING (C-14) AT GEOCHRON LABORATORIES

Krueger Enterprises Inc. Geochron Laboratories Division 24 Blackstone St. Cambridge, MA 02139

(617) 876-3691

Written by:

Charles H. Sullivan

Manager, Radiocarbon Laboratory

10 February 1984

Checked by:

Harold W. Krueger

President, Krueger Enterprises, Inc.

10 February 1984

#### RADIOCARBON DATING

Radiocarbon dating is widely used for determining the antiquity of archaeological and geological samples back to about 40,000 years ago. The measurement of radiocarbon is also useful as an indicator of the residence time of waters and as an indicator of the chemical authenticity of certain conversion of a carbon-bearing is conventionally undertaken by the conversion of a carbon-bearing sample to a form suitable for determining conversion of a carbon-bearing sample to a form suitable for determining class relative Class decay to N. Samples are converted to carbon dioxide which is either used directly or converted to methane, acetylene, or benzene depending on the form preferred by the laboratory. Gases are analyzed in proportional gas counters; benzene is analyzed in a liquid scintillation

#### RECEIPT OF SAMPLES

## LOG-IN AND REPORTING PROCEDURES

When samples are received, they are unpacked and checked against the sample list (when provided). The number appearing on the sample itself is considered authoritative if discrepancies are found. Each sample is then assigned a Geochron sample number from a roll of pre-printed sequentially numbered labels, thus eliminating any chance of two different samples receiving the same number.

Information on each sample is then entered on computer and includes the following:

~~~	GX-XXXXX (lab number)	
	Customer name and affiliation	
	Date received	
	Customer reference	
	Sample description	
	Type and condition of grant	
	nature of chemical pretreatment to	_
	applied (if any)	e

This information is stored on magnetic disk and hardcopy is printed for use as a lab data reference sheet.

All customer-submitted papers, the lab data sheets, and the lab number labels are stored in a folder labeled with the customer's name. This folder is placed with the samples in a container which is labeled on its exterior with the lab numbers of the samples, the customer's name, and the date of receipt. The container is then placed in a storage area to await performed. A card listing the customer, type of analysis to be sample control board in the main lab office.

As the analyses are completed, the results are entered on the data reference sheet. Computations are made and the final results are printed out on a standard form. All pertinent data are included on this form, which is self-explanatory. The customer folder is then sent back to the office for processing. The original report is proofread against the reference sheet by the office staff. Five photocopies of the completed standard form are made: one is sent to the customer with the original; one customer's file; and three are filed sequentially by Geochron sample number in archives stored, for safety, in three separate locations.

The original report, one photocopy, any correpondence, and the invoice are sent together to the customer by first class mail. The file containing the customer's name and affiliation.

#### **PRETREATMENT**

Samples are generally processed in order by lab number. When a sample is due for processing the customer's sample identification is again checked against our lab number. The sample then proceeds through pretreatment identified only by its GX-xxxxx (lab-number) label.

The nature of the pretreatment applied for contaminant removal depends mainly on the type of sample material. Our pretreatments are essentially the same as those used by many other radiocarbon dating labs. In general, most organic samples from archaeological or geological contexts are given sequential treatment with dilute acid and dilute base solutions in order to remove, respectively, contaminant carbonates and humates.

Certain materials, such as barium carbonate precipitated from groundwater, need no pretreatment and are analyzed directly.

### PREPARATION OF COUNTING GAS

Samples are converted to carbon dioxide by combustion in pure oxygen or by acid hydrolysis. The carbon dioxide is purified and collected by passing through a series of cryogenically cooled traps. Geochron Labs analyzes samples in the form of methane which is prepared by reacting the carbon dioxide with hydrogen at 475° C in the presence of a ruthenium catalyst.

Upon completion of the reaction the methane is purified and separated from excess hydrogen and water of reaction by passing through a series or cryogenic traps which remove water, trap methane, and allow the hydrogen to be vacuumed away. The methane is then transferred to a vacuum flask for storage of the sample gas. The flask is labeled with the lab number, the customer name, dates of reaction, and the volume of methane. The flask is proportional counters.

## DETERMINATION OF C 14 ACTIVITY

Samples of methane are counted in proportional counters especially designed for the detection of C<sup>14</sup> decay events. The counters are shielded in lead and are surrounded by annular guard counters operating in anticoincidence with the sample counters in order to reduce external radiation background to very low levels.

The sample is counted for  $C^{14}$  beta activity for at least 1200 minutes, longer if activity is very low.

Ages are calculated by comparing the beta activity of the sample to that of the international standard, NBS oxalic acid (SRM-4990). Calculations used are identical to those outlined in Stuiver and Polach, "Reporting of 14°C Data", Radiocarbon, 19(3):355-363, 1977.

Utilizing the accepted half-life of  ${\rm C}^{14}$  of 5570 years the age is calculated as follows:

AGE = (mean-life of 
$$C^{14}$$
) \* In Sample activity

Standard activity

where the mean life of  $C^{14}$  is 8036 years and the standard activity has been normalized to the assumed activity of wood formed in the years A.D. 1950.

The percent modern notation reported for geochemical samples is calculated simply as:

The error assigned to the age assessment takes into account the uncertainty due to the random nature of C14 decay in the sample, the error due to background count fluctuations in the proportional counters, and the precision with which the activity of the standard is known.

## BACKGROUND, STANDARD, AND C13 MEASUREMENTS

Measurement of the background radiation flux in the counters is necessary in order to account for detected radiation which is not due to  $C^{14}$  decay events. The background count-rate is determined by counting a methane sample prepared from very old marble which no longer contains detectable  $C^{14}$ . A background measurement is made in each counter approximately every eighth day.

Standard activity is determined by periodic counting of methane prepared from the original NBS oxalic acid standard (SRM-4990) or the newly issued replacement standard, NBS Research Material RM-49.

The value used for the modern standard in the age calculation is actually 95% of the observed activity of the standard normalized for isotopic fractionation effects to a delta  $C^{13}$  value of -19% owrt PDB.

Precise dating requires that the  $C^{14}$  activity of the sample be corrected for possible isotopic fractionation. This is also done by measuring the  $C^{13}/C^{12}$  ratio and correcting the  $C^{14}$  content accordingly. The fractionation of  $C^{14}$  relative to  $C^{12}$  is considered to be twice that of  $C^{13}$  relative to  $C^{12}$ . Samples are normalized to a  $C^{13}$  value of -25% wrt PDB as follows:

Samp. activity = Samp. activity 
$$*(1-\frac{2(25+\text{sample delta }C^{13})}{(\text{uncorrected})}$$
 (uncorrected)

\*\*  $C^{13}$  measurements are made on a small aliquot of the sample gas by analysis in a mass-spectrometer. The results are reported in delta notation relative to a standard known as PDB. The calculation of delta values is as follows:

delta 
$$c^{13}$$
 sample o/oo =  $(\frac{c^{13}/c^{12} \text{ sample}}{c^{13}/c^{12} \text{ PDB}})$  \* 1000

where the ratio of  $C^{13}/C^{12}$  in the PDB standard is 0.011237.

#### AGE REPORT FORM

The radiocarbon age of each sample is reported on a standard form (see Appendix 1). All pertinent data is included on the form and is self-explanatory. Two copies of this report are sent to the customer, one copy is placed in the customer's file, and three copies are bound in permanent logs and stored at Geochron and other locations for safety.

The customer's file containing all documentation is then filed by customer name or affiliation in a central file system.

Any excess sample is labeled with its lab number and is archived for possible future reference.

# LABORATORY PROCEDURES USED IN STABLE ISOTOPE RATIO ANALYSES (SIRA)

AT GEOCHRON LABORATORIES

21 March 1987

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General Manager

Approved by:

Harold W. Krueger

President

#### Introduction

Stable Isotope Ratio Analysis (SIRA) is the precise measurement of the abundance ratio of two stable isotopes of a particular element. Many elements have two or more naturally occurring stable isotopes. Since the isotopes of a particular element have identical chemical properties, but slightly different physical properties, they may enter into chemical reactions at different rates. As a result, natural processes have induced large local variations in the isotopic compositions of some elements, such as hydrogen, carbon, nitrogen, oxygen and sulfur. Measurement of these variations can give information about geological processes or about the origin of the material analyzed.

The basic methods of sample handling, documentation, instrumental analysis, calculation, and record keeping are similar for all types of SIRA performed at Geochron Laboratories, and can therefore be discussed in general terms. The major differences lie in the methods of sample preparation for the individual elements to be analyzed. These will be discussed separately.

#### Documentation of Samples

When a sample is received at Geochron Laboratories, the letter of transmittal is first stamped with the date received. The client's sample number, attached to the sample, is checked against the letter of transmittal, whenever possible, to insure proper agreement. The sample is then given a laboratory sample number in Geochron's sequential sample numbering system. The numbers for SIRA samples are preceded by the letter "R" which indicates that the samples are for SIRA. The "R" prefix is further preceded by another letter, or letters, denoting the particular elements for which SIRA is to be performed; "H" for hydrogen, "C" for carbon, "N" for nitrogen, "O" for oxygen, and "S" for sulfur. For example, the prefix "SR-" denotes a sample for sulfur SIRA, while "COR-" denotes a sample for both carbon and oxygen SIRA.

The SIRA number is affixed to the sample container. Thereafter, during all subsequent processing, analysis, and storage of the sample only the Geochron number is used. At the time the sample is assigned a laboratory sample number, it is also logged in on a computer generated work sheet. The information listed on the sheet at this time includes the client's name and address, the date the sample was received, any reference provided by the client (purchase order no., job number, etc.), the client's sample number, the Geochron sample number, and a description of the sample type. The work sheet is printed out, and a copy of the sheet is stored on a computer disk. The work sheet is used for recording the analytical data. The stored copy of the work sheet will be used later as the basis of the final analytical report.

A work file is also made up at this time. This file is labelled with the client's name and address, and contains all the pertinent paperwork received with the samples as well as the sample work sheet, or sheets. This file is kept with the samples throughout the analytical process until all work is completed.

#### Analytical Procedures

The general analytical procedures and techniques used at Geochron Laboratories are similar to those used by most other laboratories around the world which are engaged in SIRA work.

#### Sample Preparation

Many of the samples received for SIRA work are immediately suitable for the preparation and analysis of the particular element required. In some instances a particular mineral or material must first be separated from a bulk sample. Because of the large number of possible bulk sample materials, and the various components that may be separated from them, it is not feasible to detail all the separation procedures here. Suffice it to say that there are usually no great difficulties along these lines. Where problems do arise, they are most often due to insufficient bulk sample size, or to the general unsuitability of the particular sample material.

All samples are analyzed in gas form. Hydrogen and nitrogen samples are analyzed in molecular form. Carbon samples are analyzed as carbon dioxide. Oxygen samples are analyzed as carbon dioxide if they are from carbonates or water, and as molecular oxygen if they are from silicates. Sulfur samples are analyzed as sulfur dioxide.

Hydrogen: Hydrogen is generated from water samples by reacting about 5 microliters of sample with uranium metal at 800°C. Hydrogen from natural gas components (e.g. methane) is produced by reacting several ccSTP of the gas with wire copper oxide in a sealed glass tube at 550°C. The water of combustion is cryogenically separated from the other reaction products, and is collected and stored in a small sealed glass tube until it can be analyzed for its' hydrogen isotopic composition

Carbon: Samples of organic carbon (petroleum, wood, etc.) are combusted at 850°C in a pure oxygen atmosphere. The gases are recirculated over CuO, also at 850°C, for 10 minutes to insure complete combustion. The carbon dioxide is then cryogenically separated from the other products of combustion and the remaining excess oxygen. Organic samples with high nitrogen contents are not suitable for combustion in the recirculating system. Instead, a few milligrams of sample is flame sealed, under vacuum, in a pyrex tube containing about 1 gram of clean wire copper oxide. The tube is then oven heated at 550°C for several hours. After cooling the combustion products are withdrawn and separated cryogenically. In addition to the carbon dioxide produced by the closed tube combustion, molecular nitrogen may also be separated for analyses, as well as the water of combustion for hydrogen analyses.

<u>Nitrogen:</u> Nitrogen from organic samples is prepared by the same closed tube combustion process that is used for carbon samples.

Oxygen: Carbon dioxide is generated from carbonate samples by reacting the carbonate in vacuuo with 100% phosphoric acid at  $50^{\circ}\text{C}$ , and the evolved  $\text{CO}_2$  is purified cryogenically. Samples of water oxygen are prepared by equilibrating 2 ml of the water sample with 5 ccSTP of pure carbon dioxide gas in a sealed tube at  $25^{\circ}\text{C}$ ; the equilibrated  $\text{CO}_2$  gas is then withdrawn and cryogenically purified. Oxygen is liberated from silicate samples by reacting 10 milligrams of silicate with excess bromine pentafluoride in a nickel vessel at  $600^{\circ}\text{C}$ . The molecular oxygen produced is purified cryogenically.

Sulfur: Sulfide and native sulfur samples are combusted in a stream of flowing oxygen at 1300°C, and the resultant sulfur dioxide gas purified cryogenically. Sulfate samples are reacted with a boiling mixture of hydrochloric, hydriodic and hypophosphorous acids to release hydrogen sulfide gas, which is passed through a solution of cadmium acetate to precipitate cadmium sulfide. The cadmium sulfide is filtered, washed and dried, then combusted as any other sulfide sample. Sulfur from organic samples (e.g. crude oil) is combusted in a stainless steel bomb in the presence of excess oxygen. The sulfur dioxide produced by combustion combines with water and hydrogen peroxide in the bottom of the bomb to produce sulfuric acid, and to chemically separate it from the great excess of carbon dioxide also produced by the combustion. The sulfur is precipitated from the sulfuric acid as barium sulfate, and the sample then processed as any other sulfate.

#### **Analyses**

The gas analyses are performed on specially designed dual-inlet, double or triple collecting isotope ratio mass spectrometers. At the present time Geochron Laboratories uses a VG Isogas 602D Siamese double collecting instrument and a VG Isogas 903 triple collecting instrument. The 602D is used for all hydrogen analyses, and can also be used for carbon dioxide, nitrogen and sulfur dioxide analyses. The 903 is used for carbon dioxide and sulfur dioxide analyses, as well as for molecular nitrogen and oxygen analyses.

In practice, the actual ratio of the isotopes of interest is not measured directly, but rather the isotope ratio in the sample gas is measured in comparison to that of a working reference gas of known isotopic composition. This method of analysis avoids many possible sources of instrumental error, since they are cross-cancelling, and allows small variations in the compositions of the isotopes of interest to be measured with great precision and accuracy.

The final SIRA results are reported in terms of the relative difference (delta value, denoted by the symbol 8) in parts per thousand (termed per mil, and denoted by the symbol o/oo) of the isotope ratio of the sample compared with that of an internationally accepted standard of defined isotopic composition.

The delta values are calculated by means of the equation:

$$8 = \frac{R_{sample}}{R_{standard}} - 1 \qquad X \quad 1000$$

where  $R_{\mbox{\scriptsize sample}}$  is the isotopic ratio of the sample and  $R_{\mbox{\scriptsize standard}}$  is the isotopic ratio of the standard.

For hydrogen the isotopic ratio of interest is DH/HH, where D denotes deuterium, the hydrogen isotope of mass 2, and H denotes hydrogen, the isotope of mass 1. The ratio given here is for the "double atom" since hydrogen analyses are made on the gas in molecular form. The standard against which the calculations are made is Standard Mean Ocean Water, with a defined double atom ratio of 0.000316.

For carbon the isotopic ratio is  $^{13}\text{C}/^{12}\text{C}$ , and the standard is PDB, with a defined  $^{13}\text{C}/^{12}\text{C}$  of 0.011237.

 $14_N 15_N / 14_N 14_N$ , the standard is Air nitrogen and the double atom ratio of the standard is .007353

For exygen the isotopic ratio is the double atom ratio  $16_018_0/16_010$ , the standard is Standard Mean Ocean Water with a double atom ratio of 0.0039948.

For sulfur the ratio of interest is  $3^4 \text{S}/3^2 \text{S}$ , the standard is Troilite (meteoric sulfide) and the ratio of the standard is 0.0450045.

The standard against which each SIRA analysis is reported by Geochron Laboratories is noted at the bottom of the report form, along with its defined isotopic composition and the delta equation previously described.

The analytical data from the mass spectrometer is automatically recorded on paper tape by a desk-top computer which is an integral part of the instrument. This tape is labelled with the Geochron sample number for the sample, the working reference against which the analysis was made, the date of the analysis, and the initials of the analyst. All calculations are also performed by the computer, using programs stored in it, and the results are also recorded on the tape. The final analytical result for each sample is entered on the work sheet at the completion of each analysis.

When the analytical work is completed, the "original" of the report, stored on computer disk, is recalled into the computer and the information from the work sheet is added to it. The

final report is then printed out on the appropriate Geochron Laboratories report form, and re-checked for accuracy before being forwarded to the client, along with the invoice for the work performed. A photocopy of the final report, the laboratory work sheet, a copy of the invoice, and all copies of original paperwork that were received with the sample, or samples, are retained in the clients' permanent file. Another photocopy of the final analytical report is also kept in a permanent sequential report file.

Excess sample material which can be conveniently stored without special provision for preservation is permanently stored. Even when the client requests the return of excess material, a small amount is usually retained for possible future reference.

## RECEIVED

AUG -5 1991

## Golder Associates

LABORATORY PROCEDURES USED IN TRITIUM ANALYSES AT GEOCHRON LABORATORIES

20 August 1990

Krueger Enterprises, Inc. Geochron Laboratories Division 24 Blackstone Street Cambridge, MA 02139

> (617) 876-3691 FAX: (617) 661-0148

Written by:

Richard H. Reesman

General Manager

Checked by:

Harold W. Krueger

President, Krueger Enterprises, Inc.

#### TRITIUM ANALYSES

Tritium analyses of water are used to study groundwater systems and as a general guide to the age of the water. Natural tritium levels are low and tritium analyses are reported in Tritium Units (TU). 1 TU is 1 tritium atom in 10 atoms of hydrogen. In one liter of water, 1 TU yields 7.1 disintegrations per minute (dpm). The other unit commonly used for reporting tritium activities is the picocurie. One picocurie is 2.22 dpm. Therefore, 1 TU is the equivalent of 3.2 picocuries per liter of water. Since methods that yield measurements in picocuries usually have a lower limit of a few hundred picocuries, it can be seen that the activities of almost all natural waters will be below the background of most systems yielding picocurie measurements.

At present the tritium levels of rainfall waters are between about 10 and 25 TU, and are somewhat variable at each geographic location. The half-life of tritium is 12.26 years. Therefore, without a very good estimation of the actual rainfall tritium level at the time a particular meteoric water was precipitated, an accurate water tritium age cannot be determined. Further, at the height of hydrogen bomb testing in the early 1960's the atmospheric tritium levels in the northern hemisphere reached about 2200 TU. While atmospheric tritium activities are now approaching their pre-bomb levels, a sample of meteoric water that has been isolated since the early 1960's will still have a tritum level of almost 500 TU. Only a very slight addition of an early 1960's groundwater to a more recent one would be necessary elevate the tritium activity of the mixture to an unnaturally high level.

#### RECEIPT OF SAMPLES

#### LOG-IN AND REPORTING PROCEDURES

When samples are received, the numbers on the samples are checked against the client's written sample list. If minor discrepancies are found, the number appearing on the sample is taken as the correct one. If major discrepencies are found the client is contacted by telephone before any sample processing begins. Each sample is then assigned a Geochron laboratory number. Hereafter, all handling and processing of the sample are done using the Geochron laboratory number.

Information on each sample is then entered into a computer file, and includes the following:

- 1. Geochron Laboratory No. (T-xxxx)
- 2. Customer name and affiliation
- 3. Date received
- 4. Client's sample number

This information is store on magnetic disk and a hardcopy is also printed out for use as a laboratory reference.

All client-submitted paperwork, the laboratory reference sheets, and the samples are then placed in a container to await processing. A card listing the client, type of analyses to be performed, date received, and the Geochron laboratory numbers is placed on a sample control board.

When the analyses are completed, the final data calculations are made and entered on the laboratory reference sheet. The final results are entered into the computer file and the client's report form is printed out. Five photocopies of the original report form are made. One copy is sent to the client along with the original, one copy is included with all the other paperwork in the client's permanent file, and three copies are filed sequentially by laboratory number in Geochron's permanent archives.

The original report and one photocopy, any cover letter, and the job invoice are forwarded to the client by first class mail. The client file containing all paperwork submitted by the client, the laboratory reference sheets, a photocopy of the final report, and a copy of the job invoice are placed in the client's permanent file and stored in an alphabetical system by client name and affiliation.

#### SAMPLE PROCESSING AND ANALYSIS

Water samples are analyzed for their tritium contents by direct liquid scintillation counting. Twenty milliliters of each water sample is vacuum distilled prior to analysis. This distillation removes salts from the waters, and eliminates spurious counting effects that may occur from the deposition of salt on the walls of the counting vials.

Ten grams of the distilled sample water is weighed into a 22 ml counting vial on an analytical balance. To this is added 10 grams of Zinsser brand "Quickszint 400" scintillator. The water-scintillator mixture is homogenized on a vibratory mixer and then placed into the refrigerated counting compartment of the liquid scintillation counter. The sample is allowed to stabilized for at least 24 hours before counting begins. This stabilization period allows the sample to become fully temperature adjusted, and it also allows for the decay of any chemiluminescence that may have been induced during mixing and shaking of the sample-scintillator mixture.

Counting is performed in a Wallace-LKB Quantulus 1220 liquid scintillation counter held at 13°C, which has been determined to be the optimum counting temperature for the scintillation mixture used. Because counting is done directly on the water, and without the use of any type of enrichment process, counting rates are low for almost all natural waters. Each sample must therefore be counted for 2000 minutes to achieve the desired minimum counting uncertainty of about 2 T.U. Normally, 3 to 5 samples are counted at one time, along with either a background or standard sample. The samples are counted sequentially in 25 minute intervals until a total counting time of 2000 minutes has been accumulated for each one.

#### BACKGROUND, STANDARD AND SAMPLE TRITIUM MEASUREMENTS

Measurement of the liquid scintillation counter background is necessary to account for measured activity in the tritium activity range which is not due to sample activity. The background count rate is determined by periodically counting a "dead" sample that has been determined to contain no measurable tritium activity. The background sample employed by Geochron Laboratories is a sample of water from a deep well at Dulles Airport in Virginia. This sample was obtained from the U.S. Geological Survey and is used as a background monitor in their laboratory at Reston, Va.

Measurement of a standard activity is necessary in order to convert the measured sample tritium activities to Tritium Units (TU). The tritium standard employed by Geochron Laboratories was obtained from the National Bureau of Standards (NBS SRM 4361-94). This is a tritiated water sample whose tritium activity was carefully measured and certified by the NBS. Because tritium has a short half-life (12.26 years), each standard activity determination made at Geochron Laboratories must be corrected for the time elapsed since the NBS calibration before the correct activity-to-TU conversions can be made.

Sample tritium measurements are made in the same manner as the background and standard measurments. After subtraction of the background rate, the measured sample activities are converted to TU, using the conversion factor determined from the standard measurements and taking into account the exact sample weights used.



## Accu-Labs Research, Inc.

4663 Table Mountain Drive Golden, Colorado 80403-1650 (303) 277-9514 FAX (303) 277-9512

To:

Ms. Diane Crawford

Golder Associates, Inc.

From:

Cathy Shugarts Con

Quality Assurance Manager, Accu-Labs Research, Inc.

Re:

QA/QC Plan

Date: June 26, 1991

Enclosed is a copy of the Quality Assurance/Quality Control Plan developed by Accu-Labs Research, Inc. Please sign and date the enclosed document distribution form (F8418 Rev. \_\_) acknowledging receipt of this document. Return the form to me to ensure receipt of updates to this manual.

I encourage you to utilize the information provided and contact me directly in the event you have questions or concerns related to the QA/QC program at Accu-Labs.



## Accu-Labs Research, Inc.

4663 Table Mountain Drive Golden, Co (303) 277-9514

Golden, Colorado 80403-1650 FAX (303) 277-9512

June 26, 1991



Ms. Diane Crawford Golder Associates, Inc. 4104 148th Ave. N.E. Redmond. WA 98052

Dear Ms. Crawford,

As requested by Kathy Smit at Chen Northern, I am forwarding to you a copy of the Laboratory QA/QC Plan in use at Accu-Labs Research. Additional information related to certification and performance evaluation of the Radiochemistry Laboratory is also included.

Please contact me if you have any questions or require additional documentation.

Sincerely,

Cathy Shugarts

Quality Assurance Manager

CS/dh Enclosure

#### April, 1991 QA/QC Plan Distribution

Controlled Document Number: 58

Date Issued:

June, 26, 1991

Issued To:

Diane Crawford

Golder Associates, Inc.

Received by:

Date Received:

PLEASE RETURN THIS FORM TO:

THE QUALITY ASSURANCE MANAGER ACCU-LABS RESEARCH, INC. 4663 TABLE MOUNTAIN DRIVE GOLDEN, COLORADO 80403-1650

#### ACCU-LABS RESEARCH, INC.

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LABORATORY QUALITY ASSURANCE/QUALITY CONTROL PLAN APRIL, 1991

QA Approved by:	Cather Shermarto	<u> </u>
;	Cathy Shugarts / QA Manager, ALR	Date
•	QA Mańager, ALR	
Reviewed and	a	
Approved by:	WR Milyrun	5-10-91
	William R. Gilgren President, ALR	Date
	President, ALR	

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#### I. QUALITY ASSURANCE PROGRAM

The principal objective of the Quality Assurance Program at Accu-Labs Research, Inc. (ALR) is to provide a product of documented quality which fulfills the requirements of each client's project. The QA program was developed to follow the intent of Title 10, Code of Federal Regulations, Part 50, Appendix B (10 CFR 50). The regulation specifies the quality assurance requirements relating to work performed in support of nuclear power facilities. The quality assurance program is utilized by both laboratory management and clientele as a means of reviewing analytical results for accuracy and reliability.

The purpose of this Quality Assurance Manual is to provide an overview of the QA program. In no way does it supersede any of ALR's standard operating procedures.

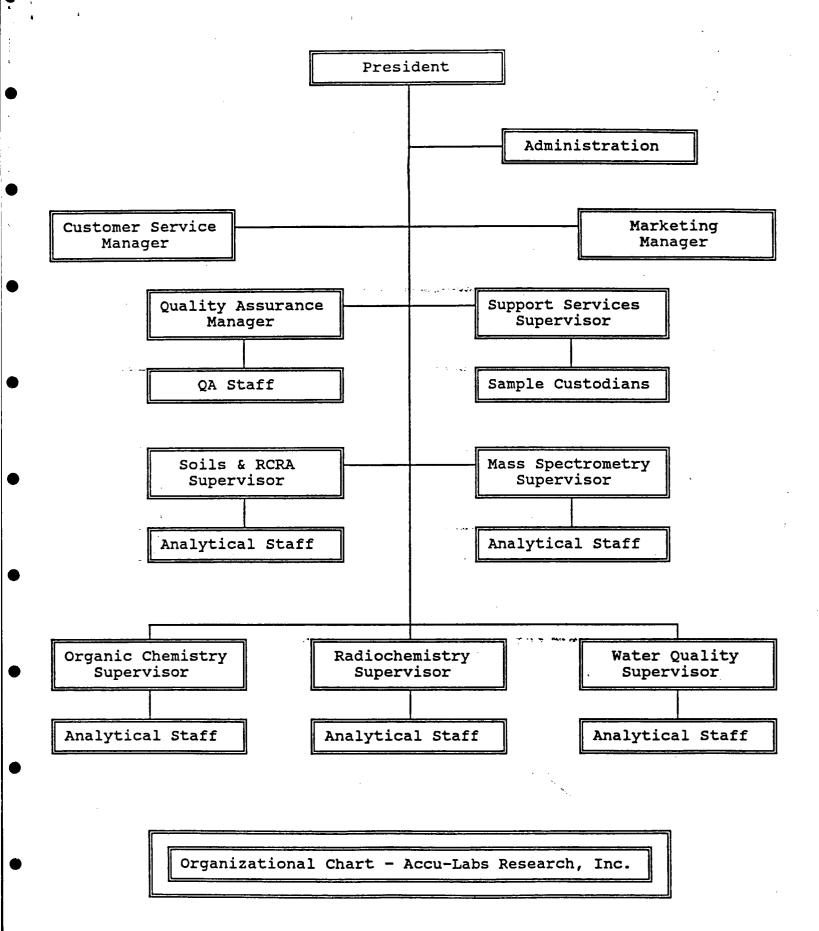
#### II. ORGANIZATION

#### LABORATORY HISTORY

Accu-Labs Research, Inc. was founded in 1971 exclusively as a Spark Source Mass Spectrometry Laboratory. The corporation diversified the laboratory operations by adding analytical capabilities in the environmentally oriented areas of Water Quality (1972), Radiochemistry (1973) and Fuels Chemistry (1974).

The laboratory was purchased by Camp Dresser & McKee (CDM) in 1976 and expanded into the areas of Soil and Overburden (1977) and Organic Chemistry (1982).

In 1984 the management of the laboratory purchased the lab from CDM, thus forming a privately owned, Colorado-based small business. The laboratory currently specializes in chemical analyses in the areas of Spark Source Mass Spectrometry, Water Quality, Radiochemistry, Soils and RCRA and Organic Chemistry.



#### STAFF POSITIONS

At the present time, Accu-Labs Research employs approximately 75 people.

Corporate policy encourages promoting from within to fill vacant positions.

Analytical staff positions are divided in two categories: Laboratory Scientists and Laboratory Technicians. Each category is further broken down into various grades according to education and experience.

Laboratory Scientist requirements are as follows:

Level I Bachelor degree; No scientific experience

Level II Bachelor degree; 1 year experience

Level III Bachelor degree; 3 years experience

Level IV Bachelor degree, M.S. preferred; 5 years experience

Level V Bachelor degree, M.S. preferred, Ph.D. beneficial; 8 years experience

Laboratory Technician requirements are as follows:

Level I No previous scientific experience necessary

Level II High School diploma or GED; No experience

Level III High School diploma or GED; 2 years experience

A position description exists for each member of the managerial and support staff which specifies the education and experience requirements, as well as the duties and responsibilities associated with the position.

Appendix Exhibit A: Abbreviated Resumes of Key Personnel

#### III. FACILITY

Accu-Labs Research maintains a single laboratory facility located in Golden, Colorado. The 27,000 square foot facility was designed explicitly as an analytical laboratory. The ground breaking ceremonies were held in April, 1990 and the occupation of the building was completed in January, 1991.

Specialized systems were installed for security, ventilation, heating and air conditioning, deionized water, fire prevention, chemical storage and laboratory waste storage. The facility is licensed by the Colorado Department of Health - Radiation Control Division - to handle radioactive materials. The license regulates the quantities of radioisotopes permitted on site.

Appendix Exhibit B: Laboratory layout

#### REFRIGERATION

The walk-in refrigerated area for sample storage is 17,000 cubic feet. The area is cooled by two compressors, each of which is capable of handling two thirds of the total cooling capacity. The temperature is maintained at  $4^{\circ}\text{C} \pm 4^{\circ}$  and is monitored daily at two locations within the refrigeration unit using thermometers calibrated against a NIST traceable thermometer. In addition, a chart recorder registers the temperature on a 24 hour, seven day cycle.

An audio/visual alarm is located at the entrance to the unit. The warning light and buzzer signal are activated when the interior temperature registers outside the preset high/low limits.

#### HOODS AND VENTILATION

The laboratory has over 130 linear feet of exhaust hoods and 24 linear feet of vented cabinets. The air flow in each hood can be adjusted for optimum usage. An alarm is activated if the flow rate is 25 ft./minute less than the selected rate. The ventilation system has been designed to prevent the recirculation of air from the sample and standard prep laboratories by using single pass air. The instrument rooms have been equipped with independent heating and air conditioning controls.

#### DEIONIZED WATER SYSTEM

The system which supplies the laboratory with deionized water is designed to produce Type II water as defined by ASTM.

The incoming water passes through a carbon filtration tank for removal of organic contaminants. A five micron filter removes any carbon particulate from the water which may have escaped the filtration tank before the water is passed through a series of cation, anion, and mixed-bed ion exchange resin tanks. The resistivity of the water is monitored after the exchange resins by means of an in-line resistivity meter. The purified water is stored in a 150 gallon tank and is passed through a final mixed-bed ion exchange polishing tank before it is ultimately provided to the laboratory.

The deionized water travels continually throughout a main pipe network for the laboratory via a recirculation pump. The circulation loop takes the purified water from the storage tank, through the polishing tank and a resistivity meter, to the labs. Unused deionized water is returned to the storage tank for future use. This system prevents the stagnation of water once it has been purified, thus ensuring water of the required purity.

A computer is used by the water conditioning company to monitor the status of the system on a daily basis. An auto-sentinel has been installed which permits direct access to information about the condition of the tanks and the need for their replacement by service personnel.

The conductivity of the deionized water is also monitored daily by an Accu-Labs Research employee at the exit point from the final polishing tank using an inline analog resistivity meter.

#### SECURITY

Access to the laboratory is monitored via a computerized key system. All employees are issued a uniquely coded key which is required for admittance into the laboratory. A computer tracks all employees who have gained access to the secured area. Each time an employee leaves the secured section of the laboratory, keyed access is required for reentry. The only area of the laboratory for which keyed access is not required is in the administrative office area during routine business hours. All other areas of the lab are secure at all times.

Visitors may be escorted through the secured laboratory area only after signing into the Visitors Log at one of the two main entrances to the laboratory. A badge is issued which must be worn by the visitor at all times while in the secured area.

#### FIRE PREVENTION AND SAFETY FEATURES

The building has been designed to minimize damage in case of fire. The floors, exterior walls and roof are concrete. Smoke and heat detectors are installed throughout the building. Fire extinguishers are located in all the laboratory areas and corridors. Sprinkler systems are in place in the acid, chemical, solvent and the laboratory waste storage areas. Moreover, the latter two storage areas are explosion proof. The floors of these rooms have been constructed

to contain chemical spills without the possibility of seepage to adjacent areas. Records are archived in an area with smoke and heat detectors. The alarm system will automatically notify the fire department in the event of a fire.

#### INSTRUMENTATION

Accu-Labs Research owns and maintains sufficient instrumentation to adequately fulfill the needs of the client. The major instrumentation includes:

Quantity	<u>Description</u>
1	Inductively Coupled Argon Plasma Emission Spectrophotometer (ICAP)
2	Graphite Furnace Atomic Spectrophotometers
1,	Flame - Atomic Absorption Spectrophotometer
1	Atomic Absorption Spectrophotometer equipped with hydride generator and cold vapor apparatus
4	UV-VIS Spectrophotometers
1	Ion Chromatograph
1	Total Organic Carbon Analyzer (TOC)
1	Total Organic Halide Analyzer (TOX)
2	Technicon Auto-Analyzer Systems
6	Selective Ion Analyzers
3	Gas Chromatograph/Mass Spectrometer/Data Systems; two equipped with purge and trap (GC/MS/DS with P&T)
1	A Gas Chromatograph with purge and trap (Hall and FID detectors)
3	Gas Chromatographs with various detectors (FID, ECD, TCD, NPD)
1 .	High pressure liquid chromatograph (HPLC)
8	Zero headspace extractors
4	Proportional Counters (10 detectors)
2	Liquid Scintillation counters

<u>Quantity</u> <u>Description</u>	
1	Gamma Spectrometer (2 detectors)
3	Radon Emanation Counters
2	Alpha Spectrometers (24 detectors)
2	Spark Source Mass Spectrometers
1	Infrared Spectrophotometer

#### IV. SAFETY

Laboratory safety is a major concern at Accu-Labs Research. A quarterly safety inspection is conducted by the Safety Officer. All safety violations are reported to the group supervisors for corrective action. The manager of the laboratory also receives a copy of the report.

Eye protection is mandatory throughout the laboratory areas. Lab coats and gloves are provided and their use is encouraged. Lab coats, gloves and respirators are mandatory in certain areas of the laboratory. Additional safety equipment, including fire extinguishers, fire blankets, showers, eyewash stations and first aid kits, is accessible to all lab personnel. All radiochemistry and sample receiving personnel must wear radioactivity monitoring badges. Fume hoods and vented enclosures with sashes are required to be used for sample digestion, distillation and extraction to protect analysts from analytical hazards.

As part of the Chemical Hygiene Plan, new employees are required to read the safety instructions. Safety training is also conducted for new employees to familiarize them with all the laboratory areas and to identify the safety precautions required for various equipment, instrumentation and analyses.

#### V. SAMPLES

SAMPLE COLLECTION AND PRESERVATION

The client is responsible for collecting and delivering samples to the laboratory in a timely manner in order for analyses to be completed within the required holding times. Accu-Labs Research personnel do not typically perform sampling.

After consultation with the client, the sample bottle request is initiated which indicates what type of sample containers and preservatives are required. The bottle request is submitted to Support Services where the necessary containers are prepared.

All containers are repeatedly rinsed with deionized water. A color coded label indicating the reagent used for sample preservation is affixed to the bottle, and the appropriate volume of preservative is added. The sample containers are packed with blue ice in a cooler. A chain-of-custody form and instructions for sampling are included with the shipment.

Appendix Exhibit C: EPA Recommended Holding Time and Sample Preservation Appendix Exhibit D: Sample Bottle Labels

#### SAMPLE RECEIPT

The sample custodian or other designated personnel have the responsibility of accepting samples from clients or transportation personnel in the laboratory designated sample receiving areas. Shipping containers are inspected for custody seals prior to unpacking and the observations are documented on the Sample Receiving/Log-In Form. Samples are unpacked in a hood if they are considered to be of a hazardous nature. If a chain-of-custody form is present, it is signed, thus acknowledging the receipt of the samples.

All paperwork which accompanies the samples is reviewed and the samples are inspected for breakage or any abnormalities. All observations are recorded on the Chain-of-Custody and on the Sample Receiving/Log-In Form. The samples are sorted by the identification on each container and assigned Accu-Labs Research numbers. Accu-Labs' unique numbering system groups together samples that were received as a set, as well as independently identifies each sample in the set. Should the sample custodian become aware of any problems with the samples, the department supervisor, project manager or customer service manager is notified so that the client may be contacted before the log-in process is continued.

# SAMPLE pH/RADIOACTIVITY SCREENING

As required for some projects, sample bottles are screened for radioactivity and/or proper preservation of the samples. If this screening process indicates radioactivity exceeding a predetermined level above background, the bottle is labeled "Radioactive" with yellow tape and appropriate safety precautions are observed.

For all samples requiring radioactivity screening, the pH/Radioactivity Screening Log form is completed. The information regarding the samples is recorded and all paperwork is forwarded to the department supervisor.

The acid and base preserved containers are monitored for all liquid samples requiring pH screening. This process identifies samples which may have been incorrectly preserved in the field and samples which need additional preservative. Samples which are determined to be improperly preserved are recorded in the pH/Radioactivity Screening Log, along with the pH reading. The appropriate department supervisor is informed of the results. Steps taken in the laboratory to resolve the problem are noted on the log sheet.

SAMPLE LOG-IN

The sample log-in procedure entails generation of all paperwork which initiates the analytical process. Following the laboratory identification and subsequent storage of all samples received, the appropriate department is notified of the samples requiring analyses with short holding times as detailed on the Sample Receiving/Log-In Form.

A laboratory work sheet is printed giving specific information regarding the client and the sample set received and lists all the parameters requested for each sample. The final analytical results may be recorded on this laboratory work sheet by each department. Work orders are also generated for the departments that will analyze the samples. The work orders inform the analysts of the parameters requested for each sample and enable the progress and completion of analyses on the sample set to be monitored.

A sample acknowledgment is generated to be sent to the client as a confirmation of receipt of the samples. The samples are identified along with the tests scheduled to be performed on the samples. A cross reference page is included, which correlates the client sample identification with the ALR sample identification.

All associated paperwork for a sample set is provided to the supervisor of the departments performing analyses on the samples.

Appendix Exhibit E: Laboratory Work Sheet

Appendix Exhibit F: Work Order

Appendix Exhibit G: Sample Acknowledgement Appendix Exhibit H: Sample Cross-Reference

SAMPLE STORAGE AND DISPOSAL

All samples labeled with the Accu-Labs' identification number are placed in designated areas dependent upon the sample matrix and preservative and the requested analyses. Non-preserved and all preserved samples, except nitric acid preserved, are stored in refrigerated areas. Each sample shelf location is identified by letter or number. If required, an internal chain-of-custody form is posted in a location near the samples to track the handling and location of samples throughout the analytical process.

Following completion of all analyses, the residual samples are transferred to designated storage areas. If an internal chain-of-custody form had been utilized, the transfer of samples and their final disposition is documented on this form.

Samples may be returned to the client upon completion of analyses. Unless otherwise requested by the client, non-hazardous samples are discarded thirty (30) days after the completion of the sample set. EPA, RCRA and local regulations are observed in the disposal of both hazardous and non-hazardous samples.

Appendix Exhibit I: Internal Chain-of-Custody Form

# VI. SAMPLE ANALYSES

Several aspects of the analytical process are monitored to ensure the generation of data of known and documented quality.

- All analysts must utilize approved Standard Operating Procedures for sample analyses.
- An analyst must be certified prior to performing any sample analyses.
- Reagents and standards must meet the requirements of the analytical method.
- All analytical instrumentation must be calibrated and maintained according to a specified schedule.
- All quality control requirements applicable to the analytical method must be observed.
- Adequate documentation of all phases of the analytical method must be maintained.
- Secondary review and supervisory approval of all data is required.

Specifics of each aspect are detailed in the following sections.

#### ANALYTICAL METHODS

Written procedures have been developed for all routine analyses performed in the laboratory. These procedures are adopted from accepted industry standards, such as those recommended by EPA, ASTM and AWWA.

The references used for developing the analytical methods are listed in the bibliography of the procedure. Samples which are analyzed for compliance with governmental regulations are analyzed using methods which are approved under the requirements of the regulatory program (ie. Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act, etc.).

The standard operating procedures are written according to format guidelines for the individual departments. The following outline is observed, when applicable, to incorporate all relevant information in the procedure:

- 1. Scope and Application
- 2. Summary of Method
- 3. Hazard Considerations
- 4. Sample Handling and Preservation
- 5. Interferences
- 6. Apparatus
- 7. Reagents
- 8. Procedure
- 9. Calculations
- 10. Reporting Format
- 11. Bibliography

Analytical procedures undergo revision to reflect current laboratory practice. Procedures which do not require revision must be periodically reviewed and approved for continued usage.

# PERSONNEL TRAINING AND CERTIFICATION

All personnel meet the educational standard as determined by their job classification. Position descriptions for all job classifications have been developed outlining the requirements and responsibilities of an individual serving in a specific capacity in the laboratory.

An orientation as to the specifics of the quality assurance program at Accu-Labs Research is conducted by QA Department personnel for all new analysts within the first few weeks of employment.

To be certified to perform analyses of samples for clients, each analyst must demonstrate a working knowledge of the technical and theoretical aspects of his/her specialty and position. Due to the high degree of variation in the individual departments, there are also differences in the certification processes. However, each technician is required to undergo individual training in his or her department prior to unsupervised analysis of any samples.

The training consists of at least the following points:

- 1) The trainee should become familiar with the procedures to be performed along with the reagents and equipment used.
- 2) Under the direction of a certified analyst, the trainee should analyze a set of known samples to demonstrate a good working knowledge of the analysis that will be performed.
- 3) Finally, the trainee should go through all the steps of the analysis, including the preparation of standards and reagents. When the trainee has proven competence of the specific analysis, department paperwork is completed to document the trainee's certification.

The department supervisors are ultimately responsible for the training and certification of their staff. Actual training, however, may be carried out by a designated member of the staff. Current employee certification is kept on record in each department and updated as required.

# INSTRUMENT CALIBRATION AND MAINTENANCE

Instruments and other measuring and testing devices used in activities affecting quality are controlled, calibrated and maintained periodically to assure accuracy within specified limits. It is the responsibility of the analyst using the instrument to perform and document the required calibration.

Calibration information may be documented in one of several locations. The requirements for calibration vary with each instrument, thus necessitating flexibility in the recording of such information. Calibration records are maintained by the group responsible for the equipment.

If the calibration schedule has not been observed, or the required level of accuracy cannot be attained for a specific instrument, the instrument is labeled "HOLD" and is unavailable for use until the specifications are attained. Should an instrument be found to be out of calibration, all data obtained subsequent to the last successful calibration is reviewed by the Group Supervisor and analyses are repeated as deemed necessary.

All controlled analytical instrumentation is maintained according to specified preventative maintenance schedules. Maintenance procedures are performed by either skilled analysts or contracted service personnel. All maintenance of instruments is documented in the instrument Life History Log.

#### QUALITY CONTROL

The quality control mechanisms used at Accu-Labs Research, Inc. are designed to assist in the evaluation of the data produced by the laboratory. Each department specifies the frequency of quality controls applicable to the analyses performed.

Precision is monitored by repeating at random a specified number of samples or sample spikes.

Accuracy is measured through the utilization of reference materials of known concentration (such as those available from EPA, NIST, etc.). Accuracy is dependent upon the bias, or systematic error, inherent in the analytical method or measurement system. Method and measurement system bias is evaluated by treating a reference material in the same manner as a sample, thus referred to as a laboratory control sample. For some analyses it is possible to determine measurement system bias independently by analyzing an untreated reference material, commonly identified as a calibration verification sample. System bias introduced by reagents, glassware, water purity, etc. is monitored by analysis of a reagent blank.

Sample matrix interferences may be monitored by evaluating the percent recovery (accuracy) of a standard spike added to a second aliquot of the sample prior to analysis.

The confidence limits for the quality controls have been established by each department and acceptance of the data is determined by achieving analytical results within the prescribed limits. Should these limits not be attainable, appropriate corrective action, as prescribed in written procedures, is required to remedy the situation.

# LABORATORY REAGENTS AND STANDARDS

The quality of reagents used is dependent upon the nature of the analysis.

"Analytical reagent" grade is used when no minimum requirement is stated. When necessary for a particular procedure, a higher grade reagent is obtained. High purity acids and/or solvents are used for digestions or extractions for trace level analyses. Gases utilized for analyses are of several different grades depending on analytical and instrumental requirements. The tanks are labeled according to the grade of gas in the cylinder. Whenever possible, each department routinely uses primary standards that are traceable to NIST (National Institute of Standards and Technology).

#### Chemical Log-In

Chemicals that are received in the laboratory are assigned an ALR chemical log number. Each container is tracked using a computer data base which notes the supplier, lot number, quantity received, grade of purity, expiration date and the ALR assigned chemical log number.

A label is affixed, if the size of the container permits, with the date received, date opened and expiration date. A system is in place for monitoring all chemicals currently in use and all chemicals that have been depleted. Each department maintains a supply of chemicals that are routinely used by its staff. Bulk chemicals such as acids, bases and solvents are kept in centralized chemical storage areas and are not assigned ALR chemical log numbers.

Reagent and Standard Preparation

All reagents prepared from stock chemicals have a label affixed which indicates the compound identification, chemical log number, date prepared, by whom it was prepared and, if applicable, expiration date. By using the chemical log number, the reagents are thus traceable to the chemicals from which they were made.

Appropriate volumetric glassware, pipets or syringes are used for reagent and standard preparation. All glassware is thoroughly cleaned prior to any dissolution of standard material. Reagents and standards prepared on a weight basis require the utilization of balances calibrated according to specified requirements. Standard solutions and reagents requiring refrigeration are kept in areas isolated from samples.

#### DATA REVIEW

Upon completion of the analyses, the results are calculated by the analyst either by hand or by computer program. The analyst initials the data sheets and submits the data for recalculation. The QA data and all calculations are rechecked by a second individual who initials the pages as evidence of recalculation and submits the data to the department supervisory personnel. The supervisor or assistant initials acceptable results or indicates any unacceptable results that require sample reanalysis.

All approved data are recorded on the laboratory work sheet or another approved form. Transfer of sample results from the laboratory notebook to the laboratory work sheet is performed by the supervisory staff. Order of magnitude agreement between the analyses is reviewed upon completion of all data. Should a result appear incorrect, a rerun of the sample is requested. After all data questions

have been satisfactorily resolved, the laboratory work sheet is initialed and submitted for typing. The signature on the issued report is final verification of acceptance of the data by the supervisor.

# DOCUMENTATION OF ANALYTICAL RESULTS

Adequate written records are maintained of analyses performed. All data recorded manually are written into bound paginated notebooks or on approved preprinted forms in ballpoint pen with black ink. Data notebooks are serially numbered according to department and type of analysis. Active notebooks are retained in a secure location in each department until they are archived. An instrument or computer printout or strip chart recording can be substituted for a handwritten notebook. These pages are dated, initialed and traceable to the correct sample and type of analysis.

All information pertinent to the generation of the analytical results must be documented. As required, the documentation should include the type of analysis, analyst, date of analysis, ALR sample number, QC samples, instrument identification, sample aliquot, concentration or dilution factors, calibration information and sample results. Any errors in the documentation are crossed out and initialed by the author. Correction fluid is not permitted.

Raw data and final reports are archived for a period of five years.

Appendix Exhibit J: Data Notebook Sheet

#### VII. QUALITY ASSURANCE DEPARTMENT

The QA Department functions to ensure that the clients are provided with a product of documented quality. It is the duty and responsibility of the quality assurance manager to implement and recommend measures for the realization of quality objectives. Moreover, the manager is responsible for the maintenance and review of the quality assurance program for effectiveness and currency of content. The program is scrutinized and modified if necessary to fulfill the requirements of the client. In these tasks, the QA Manager is assisted by quality assurance technicians. The Quality Assurance Department answers directly to the President of the corporation.

The QA Department personnel at Accu-Labs Research monitor the following activities:

- Laboratory certification
- Personnel training and certification
- Calibration and maintenance of instrumentation
- Incorporation of revisions to analytical methods
- Data review
- Internal and external audits
- Procedural variances
- Compliance with Federal and State regulations governing analyses of various types
- Corrective action
- Acceptance limits for precision and accuracy
- Traceability of standards
- Recommended sample preservation and storage requirements

Quality control activities which have not been previously addressed in this manual are discussed in the following sections.

# BLIND PERFORMANCE EVALUATION AUDIT

Blind performance evaluation samples which contain analytes of known concentration are used to determine the routine performance of the various departments. These performance evaluation samples are prepared or purchased by the QA Department and are submitted blindly to each department of the laboratory on a quarterly basis.

The analytical results and the associated raw data package are evaluated for accuracy and completeness by the QA Department personnel. The findings are reported in writing to the department supervisor and to the president of the corporation. The department management is required to develop and implement a plan of corrective action which is reported to the QA Department in response to the audit findings.

Performance evaluation samples are a good independent indicator of laboratory performance. Clients are encouraged to submit "blinds" periodically to the laboratory along with their routine samples.

#### DOCUMENT CONTROL

Activities affecting quality are described in writing so that any individual procedure can be fully described and supported at a later date. Written documents which must be modified by following an established change order procedure are referred to as "controlled documents." Controlled documents include, among others, forms, analytical procedures, equipment specifications and calibration and maintenance procedures. Every controlled document has a unique document number.

When a document or form is modified, the document number is altered correspondingly with a revision letter suffix. The original and the revised document are kept by the QA Department in permanent files.

The Quality Assurance Department is responsible for distributing controlled documents to the appropriate activity centers. Group supervisors are responsible for distribution of these documents within their departments.

Deviations from or modifications of a documented procedure are considered variances. The variance may involve calibration of the instrumentation, meeting established quality assurance requirements, or modification of the analytical process. Documentation of the changes are noted on a variance form. If a similar type of variance for a specific procedure occurs repeatedly, revision of the procedure might be requested to incorporate the recurring variance.

The QA Department personnel monitor the corrective action implemented as a result of a variance. The original variance forms are retained in the QA Department's permanent files. Copies are enclosed in the raw data package. If deemed necessary, the client is notified of the analytical variance.

#### INTERNAL AUDITS

Departmental audits are performed in order to monitor the effectiveness of the QA program and the means by which each laboratory complies with program requirements. QA Department personnel prepare and conduct the audits which are designed to evaluate a specific aspect of the program. For example, equipment calibration and maintenance, personnel certification or the utilization of currently approved procedures may be audited on an annual basis.

Appendix Exhibit K: Variance Form

Following the audit, the findings are reviewed with appropriate departmental personnel to discuss possible corrective action to any quality problems or concerns noted by the auditors. The audit findings are reported in writing to the department supervisor and to the president of the corporation. The department management is required to develop and implement a plan of corrective action which is reported to the QA Department in response to the audit findings.

#### EXTERNAL AUDITS

The laboratory is frequently audited by clients, prospective clients and representatives of regulatory agencies. These audits are routinely pre-arranged to ensure the availability of key personnel. The QA Manager accompanies the auditors through the laboratory. The findings from these audits are reviewed with the appropriate departments and required corrective actions are implemented.

# LABORATORY CERTIFICATION

Many state and federal agencies acknowledge the need for standardization of methods as well as recognition of laboratories which meet these standards and provide a service of the highest quality. Voluntary programs have been established by which participating laboratories which successfully analyze performance evaluation samples are eligible for certification by the agency. Accu-Labs Research routinely participates in cross-check programs conducted by governmental regulatory agencies. The results of semi-annual participation in the Water Pollution and Water Supply Performance Evaluation Programs are used for certification purposes for organic and inorganic analyses by state and regional regulators.

A State may have primacy over the EPA in the certification process, if the State demonstrates a certification program which meets or exceeds the requirements of the program established by the EPA. Oftentimes states within a specific region will recognize the certification of a laboratory in another state as valid and extend certification to the laboratory by reciprocity. In states without enforcement responsibility, laboratories will be certified by the Regional Office of the EPA on an interim basis.

The Colorado Department of Health currently certifies laboratories for the analyses of contaminants covered by the National Interim Primary Drinking Water Regulations including microbiological, inorganic and organic constituents.

Accu-Labs holds certification in all of these areas. The Region VIII Office of the EPA certifies laboratories for radiochemical analyses for compliance with the drinking water regulations. Accu-Labs is also certified for all regulated radiochemical constituents.

Accu-Labs Research willingly and actively pursues certification in recognized programs in many States and EPA Regions. Recertification on an annual basis demonstrates that the quality assurance program utilized by Accu-Labs is successful in providing analytical services of the highest quality to the environmental community.

# VIII. APPENDIX

- Exhibit A. Abbreviated Résumés of Key Personnel
- Exhibit B. Laboratory Layout
- Exhibit C. EPA Recommended Holding Times and Sample Preservations
- Exhibit D. ALR Sample Bottle Labels
- Exhibit E. Laboratory Work Sheet
- Exhibit F. Work Order
- Exhibit G. Sample Acknowledgment
- Exhibit H. Sample Cross-Reference
- Exhibit I. Internal Chain-of-Custody Form
- Exhibit J. Data Notebook Sheet
- Exhibit K. Variance Form

#### WILLIAM R. GILGREN

# Laboratory Manager

Date of Birth: January 29, 1947

Education: Bachelor of Science, Chemistry, University of Nebraska.

Lincoln, Nebraska, 1969.

Experience: January 1984 to Present: Accu-Labs Research, Inc.,

formerly Camp Dresser and McKee, Inc., Analytical Services, Golden, Colorado, President and Co-Owner. Responsible for overall management of the Water Chemistry, Radiochemistry, Organic Chemistry, Soils/RCRA and Mass Spectrometry Groups, the Quality Assurance Department and the Administrative Support Group. Evaluate and coordinate staffing levels and marketing effort in relation to backlog projections. Develop annual budget including capital equipment, personnel and expense projections. Responsible for cash flow, payables and accounts receivable.

August 1976 to December 1983: Camp Dresser and McKee, Inc., Operations Manager. Responsible for the coordination of the entire laboratory operation, and for all overall data quality and client satisfaction.

November 1971 to July 1976: Accu-Labs, CDM/Limnetics, Wheat Ridge, Colorado, Supervisor, Mass Spectrometry Group. Responsible for all mass spectrometric functions, initiation and completion of laboratory work, writing reports, and photoplate interpretation. Oversaw group quality control program. Actively sold mass spectrometric analyses. In charge of material sales of related products and supervised company maintenance personnel. Responsible for project coordination.

October 1969 to August 1971: U.S. Army, First Lieutenant. Tactical Operations Officer for Infantry Battalion in Korea.

Professional Societies:

American Chemical Society
The American Society for Mass Spectrometry

#### MARY B. FABISIAK

# Customer Service Manager

Date of Birth: September 28, 1957

Education: Bachelor of Science, Biology, University of Wisconsin -

Stevens Point, Stevens Point, Wisconsin, 1979. Chemistry

Minor.

Experience: June 1990 to Present: Accu-Labs Research, Inc., Golden, Colorado, Customer Service Manager. Specialize in handling client concerns, problems and questions. Work with log-in

personnel, supervisors and marketing manager to clarify instructions, coordinate projects and supply accurate infor-

mation to all parties.

April 1988 to June 1990: Accu-Labs Research, Inc., Supervisor of Water Quality Group. Supervise and schedule a laboratory staff of twenty performing water quality chemistry. Responsible for supply and equipment purchasing, client contact, equipment maintenance and repair, writing and reviewing reports, and maintenance of quality control. Provide technical consultation to clients on analytical capabilities, and write proposals.

May 1985 to March 1988: Accu-Labs Research, Inc., Assistant Supervisor of Water Quality Group. Responsibilities included managerial and supervisory duties, routine wet chemical and instrumental analyses of water samples.

December 1984 to September 1985: Accu-Labs Research, Inc., Quality Assurance Manager. Responsible for administration of all Quality Assurance matters.

April 1984 to May 1985: Accu-Labs Research, Inc., Water Laboratory Scientist. Responsible for routine water analyses using auto analyzers, atomic absorption, spectroscopy and organic carbon analyzers.

Previous employment: Lead Geochemical Laboratory Technician, Bondar-Clegg, Inc., Lakewood, Colorado; Geochemical Laboratory Technician, Skyline Labs, Inc., Wheat Ridge, Colorado.

Certifications: Class D Water Plant Operator Certification, 1984

Class D Waste Water Plant Operator Certification, 1984 Level II Colorado Water Quality Analyst Certification, 1986

Memberships: Rocky Mountain Water Quality Analyst Association

#### THOMAS J. BALKA

#### Marketing Manager

Date of Birth: June 19, 1948

Education: Bachelor of Arts, Chemistry (Minor Biology), Metropolitan

State College, Denver, Colorado, 1978.

Experience: January 1984 to Present: Accu-Labs Research, Inc., formerly

Camp Dresser and McKee, Inc., Golden, Colorado, Marketing and Co-Owner. Marketing laboratory services, writing proposals, project management, business development, client

satisfaction and relations.

September 1985 to April 1988: Accu-Labs Research, Inc., Quality Assurance Manager. Responsible for administration of all Quality Assurance matters including laboratory procedures, test equipment and data analyses. Conduct internal audits to maintain a high level of quality control. Assist in client Quality Assurance audits of the laboratory.

February 1980 to December 1983: Camp Dresser and McKee, Inc., Laboratory Marketing. Duties same as above.

August 1978 to February 1980: Camp Dresser and McKee, Inc., Laboratory Scientist. Performed chemical analyses of water samples in general chemistry lab.

# CATHERINE I. SHUGARTS

Quality Assurance Manager Special Projects Manager

Date of Birth: March 15, 1956

Education: Bachelor of Science, Biology, Loyola University of Chicago,

Illinois, 1979.

Experience: April 1988 to Present: Accu-Labs Research, Inc., Golden, Colorado, Quality Assurance and Special Projects Manager. Responsibilities include maintenance of the system for controlling quality assurance records while monitoring adherence to the program through internal audits of laboratory procedures, equipment, data records and reports. As required, duties involve project management, methods development, instrumentation acquisition, computer assistance and report preparation of various types for administrative purposes. Supervise the activities of quality assurance technicians.

> April 1985 to April 1988: Accu-Labs Research, Inc., Supervisor for Water Quality Group. Supervise and schedule a laboratory staff of twelve performing water quality chemistry. Responsible for writing and reviewing reports, initiation and completion of laboratory work, research and development of analyses for special projects and the maintenance of quality control.

> January 1984 to April 1985: Accu-Labs Research, Inc., formerly Camp Dresser and McKee, Inc., Assistant Supervisor for Water and General Chemistry Group. Responsibilities included managerial and supervisory duties, QA/QC data quality control; and wet chemistry and instrumental analyses.

> January 1983 to December 1983: Camp Dresser and McKee Inc.. Assistant Supervisor for Water and General Chemistry Group. Duties same as above.

July 1980 to January 1983: Camp Dresser and McKee Inc.. Laboratory Scientist. Responsibilities included wet chemistry and instrumental analyses using flame and graphite atomic absorption, gas chromatography, auto analyzers, spectroscopy, and organic carbon and halide analyzers.

Previous employment: Water Laboratory Technician, Interlake Technical Center, Riverdale, Illinois.

Certifications: Colorado Water Quality Analyst Certification, Level III, 1987.

Memberships: Rocky Mountain Water Quality Analyst Association American Society for Testing and Materials

# Accu-Labs Research, Inc.

#### CONSTANCE K. HIATT

#### Supervisor - Support Services

Date of Birth: August 12, 1944

Education: Pontiac Township High School, Pontiac, Illinois, May 1962.

Experience: October 1989 to Present: Accu-Labs Research, Inc., Golden, Colorado, Supervisor - Support Services. Oversee shipping and receiving, sample bottle preparation and courier duties,

in addition to the responsibilities below.

January 1984 to October 1989: Accu-Labs Research, Inc., formerly Camp Dresser and McKee, Inc., Analytical Services, Wheat Ridge, Colorado, Senior Secretary and Sample Custodian. Responsibilities include computerized sample log-in on Envirobase system creating all necessary paperwork for analysis and sample tracking. Generate draft and numbered invoices and sample acknowledgments. Supervise assistant Sample Custodians. Assist with report generation on CPT 9286 and computerized network system, process purchase orders, order laboratory and general office supplies, and schedule courier duties.

September 1978 to December 1983: Camp Dresser and McKee, Inc., Senior Secretary and Sample Custodian. Sample log-in duties with completion of necessary paperwork consisting of control sheets, work orders, invoices, sample acknowledgments and chain-of-custody forms. Some computer data entry and assisting on CPT 8000 for report generation.

Previous employment: Secretary-Receptionist, Emmaus Lutheran Church, Denver, Colorado; Administrative Secretary, Evenglow Lodge, Pontiac, Illinois.

#### WENDELL D. FISCHER

#### Group Supervisor of Soils/RCRA Group

Date of Birth: February 14, 1960

Education: Bachelor of Science, Biological Sciences, University of

Nebraska, Lincoln, Nebraska, 1985.

Experience: May 1990 to Present: Accu-Labs Research, Inc., Golden,

Colorado, Supervisor of Soils/RCRA group. Responsibilities include supervising and managing of all laboratory activities. Technical advisor on analytical procedures and development. Responsible for client contact, control of quality data,

personnel and equipment procurement.

August 1987 to May 1990: Accu-Labs Research, Inc., Soils/ RCRA Assistant Supervisor. Duties included data compilation, maintenance of QA records and procedure manuals, scheduling of laboratory activities and client contact.

September 1985 to August 1987: Accu-Labs Research, Inc., Laboratory Scientist for Soils and Overburden. Responsibilities included pH and electrical conductivity analyses on soil samples, preparation and analyses of soil samples for trace elements by Atomic Absorption Spectroscopy. Also colorimetric, chemical, gravimetric, sulfur content and RCRA analyses of soils and overburden.

Additional Experience:

April 1989 to March 1991: Accu-Labs Research, Inc., Health and Safety Officer. Duties included the assurance of a safe working environment and administering first aid when needed. Also responsible for informing personnel of safe working habits and appropriate safety precautions, performing periodic safety inspections of the laboratory facility, maintenance of safety equipment and safety supply inventory.

#### STEPHEN R. WATKINS

#### Mass Spectrometry Supervisor

Date of Birth: August 26, 1947

Education: University of Colorado, Denver, Colorado, Graduate Studies, 1980 - 1985, 1988 - Present.

Bachelor of Arts, Mathematics, University of Colorado, Denver, Colorado, 1978.

Classes completed and certificates received: Management of Hazardous Wastes Seminar, University of Colorado, Boulder, 1987.

Hazardous Chemical Safety School, J.T. Baker, Dallas, Texas, 1978.

Experience: July 1987 to Present: Accu-Labs Research, Inc., Golden, Colorado, Supervisor of Spark Source Mass Spectrometry group. Responsible for supervision of technical personnel and scheduling work load. Duties include photoplate data interpretation, preparing reports and maintaining client contact, maintenance and repair of mass spectrometers, developing procedures, and ensuring compliance with Quality Assurance Program.

October 1985 to July 1987: Accu-Labs Research, Inc., Laboratory Scientist, Mass Spectrometry Department. Responsible for technical analyses of trace elements using AEI-702 and AEI-7 instrumentation. Other duties included preparation of samples, development of photoplates and photoplate data interpretation.

August 1983 to August, 1985: University of Colorado, Denver, Colorado, Teaching Assistant, Mathematics Department. Responsible for conducting freshman and sophomore level mathematics classes.

Additional Experience: January 1989 to March 1991: Accu-Labs Research, Inc., Hazardous Waste Officer. Responsible for the management of hazardous chemicals and wastes.

April 1987 to January 1989: Accu-Labs Research, Inc., Safety Officer.

Other Work Experience:

Quality Control Technician, Metals Laboratory, Inc., Denver, Colorado; Operations Manager, Big T Janitorial, Inc., Arvada, Colorado; Laboratory Technician to chief chemist, Gardner-Denver/Cooper Industries, Denver, Colorado.

Professional Mathematical Association of America Societies: American Society for Metals

#### CHRISTOPHER C. SHUGARTS

Group Supervisor of Organic Chemistry Group Project Manager

Date of Birth: December 7, 1950

Education: Bachelor of Arts, Chemistry (Minor Biology), University of

Colorado at Denver, Denver, Colorado, 1979.

Experience: January 1985 to Present: Accu-Labs Research, Inc., Golden, Colorado, Supervisor of Organic Chemistry Group and Co-Owner. Supervise and schedule a staff of analysts who perform all phases of organic chemical analyses. Responsible for writing and reviewing reports, initiation and completion of laboratory work, research and development for special projects and the maintenance of quality control. Provide technical consultation to clients on analytical abilities and maintain client contact. One of the senior technical advisors for the laboratory.

> January 1984 to December 1984: Accu-Labs Research, Inc., Wheat Ridge, Colorado, formerly Camp Dresser and McKee, Inc., Analytical Services, Supervisor and Co-Owner. Supervise and schedule a staff of eleven laboratory analysts doing water, soils, overburden, waste chemical and physical analyses. Responsible for writing and reviewing reports, initiation and completion of laboratory work, research and development of analyses for special projects and the maintenance of quality control. Provide technical consultation to clients. on analytical abilities and maintain client contact.

January 1981 to December 1983: Camp Dresser and McKee, Inc. Supervisor. Duties same as above.

August 1979 to January 1981: Camp Dresser and McKee, Inc., Fuels, Soil and Overburden Senior Laboratory Chemist. Preparation, instrumental and wet chemical analyses, and data compilation.

Prior experience: Undergraduate Research and Teaching Assistant, University of Colorado at Denver, Denver, Colorado.

**Professional** Societies:

American Chemical Society Rocky Mountain Chromatography Discussion Group Rocky Mountain Mass Spectroscopy Group

#### HARRY VICTOR (BUD) SUMMERS III

Group Supervisor of Radiochemistry Group Project Manager

Date of Birth: July 1, 1946

Education: Master of Science, Radiochemistry, New Mexico Highlands

University, Las Vegas, New Mexico, 1971.

Bachelor of Arts, Chemistry, University of New Mexico,

Albuquerque, New Mexico, 1969.

Experience: January 1984 to Present: Accu-Labs Research, Inc., Golden, Colorado, formerly Camp Dresser and McKee, Inc., Analytical Services. Supervisor and Co-Owner. Direct and supervisor

Services, Supervisor and Co-Owner. Direct and supervise radiochemistry group. Develop and revise analytical proce-

dures for radiochemical analyses in environmental

monitoring. Maintain and develop QA/QC program to meet state and federal requirements in coordination with QA manager. Contact clients regarding technical information and assistance. Approve and analyze analytical results generated from radiochemistry laboratory. Evaluate and schedule laboratory personnel, make equipment purchases,

develop scopes of work and price proposals.

July 1981 to December 1983: Camp Dresser and McKee, Inc., Supervisor of Radiochemistry Group. Duties same as above.

July 1980 to June 1981: Camp Dresser and McKee, Inc., Senior Chemist and Program Manager. Senior Chemist responsibilities included special analyses procedure development; development and updating of procedures required under federal regulations including Resource Conservation and Recovery Act and Toxic Substance Control Act. Primary areas of analytical responsibility was in CDM's radiochemistry department, involving radiochemistry of tissue, soils, water, and air particulate samples. Program Manager responsibilities included serving as a program manager for large multi-department analytical contracts, maintaining sound written and verbal communications, budgetary accounting, and invoicing preparation, in-house sample and communication coordination.

Positions held at Controls for Environmental Pollution, Santa Fe, New Mexico prior to joining CDM: Director of Special projects, Reactor Controls and Water Quality Divisions; Laboratory Manager; Supervisor of the Environmental Chemistry and Nuclear Measurements Division.

Professional Societies

Societies: American Chemical Society
Health Physics Society

#### EYDA M. HERGENREDER

Group Supervisor of Water Quality Group

Date of Birth: March 28, 1954

Education: Bachelor of Science, Biology (Minor Chemistry) New Mexico

Institute for Mining and Technology, Socorro, New Mexico,

1976.

Experience: June 1990 to Present: Accu-Labs Research, Inc., Golden,

Colorado, Supervisor of Water Quality Group. Supervise and schedule a laboratory staff of twenty performing water quality chemistry. Responsible for supply and equipment purchasing, client contact, equipment maintenance and repair, writing and reviewing reports and maintenance of quality control. Provide technical consultation on analytical capa-

bilities and write proposals.

May 1985 to June 1990: Accu-Labs Research, Inc., Water Laboratory Assistant Supervisor. Primary duties included analyses of metals by ICP and Graphite Atomic Absorption. Assisted in reporting of data for the EPA contract work. Monitored all atomic absorption work for both water and soil groups.

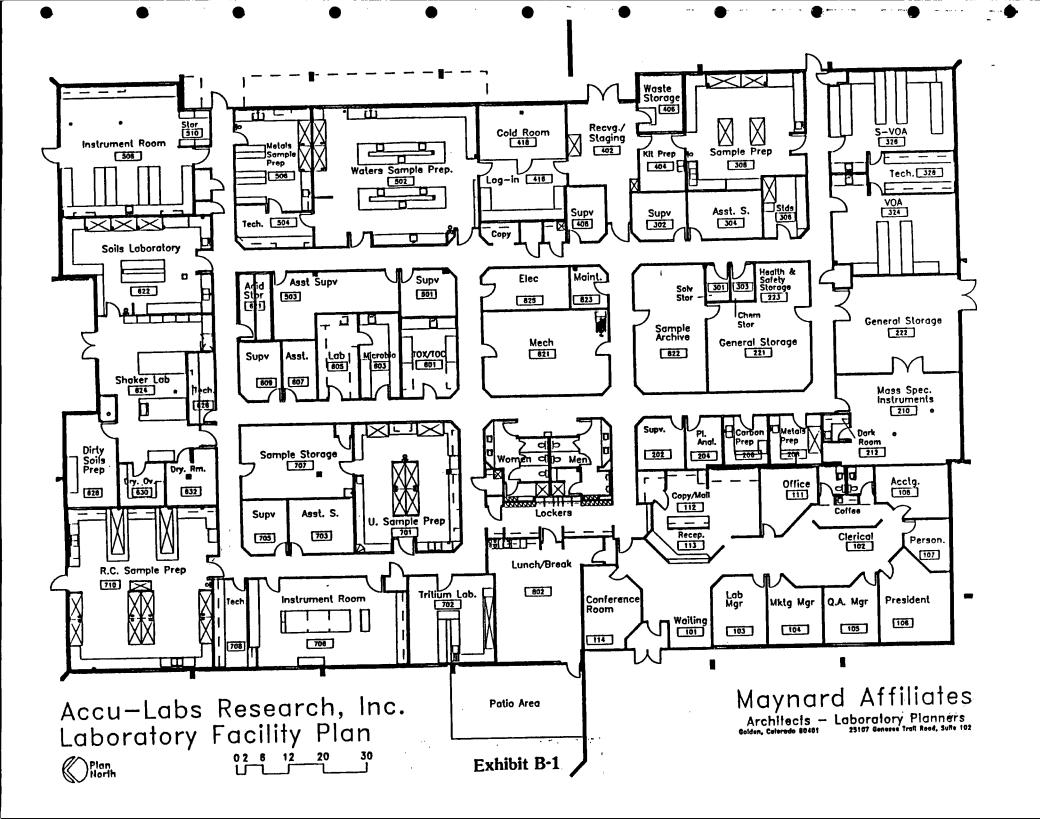
January 1984 to May 1985: Accu-Labs Research, Inc., formerly Camp Dresser and McKee, Inc., Analytical Services, Laboratory Scientist. Wet chemistry and instrumental analyses of water for all routine parameters. Instrumentation included flame and graphite atomic absorption, gas chromatography, auto analyzers, spectroscopy, and organic carbon and halide analyzers.

March 1981 to December 1983: Camp Dresser and McKee Inc., Laboratory Scientist. Duties same as above.

Prior employment: Laboratory Technician, Controls for Environmental Pollution, Santa Fe, New Mexico.

Professional

Societies: Society for Applied Spectroscopy



# EPA Recommended Holding Times and Sample Preservatives

Parameter	Container Plastic (P) Glass (G)	Preservative	Recommended Holding Time
Acidity	P,G	Cool 4°C	14 d
Alkalinity	P,G	Cool 4°C	14 d
Ammonia	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , cool 4°C	28 d
BOD	P,G	Cool 4°C	48 h
Boron	P,G	5 mL/L HNO <sub>3</sub>	6 m
Bromide	P,G	None required	28 d
COD	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , cool 4°C	28 d
Chloride	P,G	None required	<u>.</u> 28 d
Chlorine, total residual	P,G	None required	Immediately
Chromium, hexavalent	P,G	Cool 4°C	24 h
Coliforms	P,G	Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>	6 h
Color	P,G	Cool 4°C	48 h
Cyanide	P,G	Cool 4°C, NaOH to pH >12 (8 pellets/L) 0.6 g ascorbic acid <sup>1</sup>	14 d
Dustfall	Dustfall Jar	NA	NA
Filtering	P,G	0.45 micron membrane	On site
Fluoride	P	None required	28 d
Hardness	P,G	5 mL/L HNO <sub>3</sub>	6 m
Total Petroleum Hydrocarbon	G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , Cool 4°C	28 d
Iodine	P,G	Cool <4°C	24 h
Mercury	P,G	5 mL/L HNO <sub>3</sub>	28 d

# EPA Recommended Holding Times and Sample Preservatives (Continued)

Parameter	Container Plastic (P) Glass (G)	Preservative	Recommended Holding Time
Metals	P,G	5 mL/L HNO3	6 m
Nitrate	P,G	Cool 4°C	48 h
Nitrite	P,G	Cool 4°C	48 h
Nitrate-Nitrite	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , Cool 4°C	28 d
TKN and Organic Nitrogen	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , Cool 4°C	28 d
Oil and Grease	G	2 mL/L $H_2SO_4$ or 2 mL/L $HC1$ , Cool $4^{\circ}C$	28 d
Organic Carbon	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> or HCl, Cool 4°C	28 d
На	P,G	None required	Immediately
Phosphorus, total	P,G	2 mL/L H <sub>2</sub> SO <sub>4</sub> , Cool 4°C	28 d
Ortho-Phosphate	P,G	Cool 4°C, filter immediately	. 48 h
Phenolics	G	Cool 4°C, H <sub>2</sub> SO <sub>4</sub> <ph 2<="" td=""><td>28 d</td></ph>	28 d
Silica	P,G	5 mL/L HNO3	6 m
TDS	P,G	Cool 4°C	7 d
TSS	P,G	Cool 4°C	7 d
Settleable Solids	P,G	Cool 4°C	48 h
Total Solids	P,G	Cool 4°C	7 d
Volatile Solids	P,G	Cool 4°C	7 d
Total Organic Halides	G	Cool 4°C	28 d
Specific Conductance	P,G	Cool 4°C	24 h
Sulfate	P,G	Cool 4°C	28 d

# EPA Recommended Holding Times and Sample Preservatives (Continued)

Parameter	P1	ontainer astic (P) lass (G)	Preservative		Recommended Holding Time	
Sulfide		P,G	1.5 mLs 2N Zinc acetate/ 7 d liter, 1.0 mL 6N NaOH per liter, fill bottle completely, cool 4°C			
Sulfite		P,G	None required		Immediately	
Surfactants		P,G	Cool 4°C		48 h	
Temperature		P,G	None required			
Turbidity		P,G	Cool 4°C	48 h		
Pesticides, Herbicides, and PCB's	t	with eflon ned cap	Cool 4°C	7 d until extrac tion, 40 d after extraction		
Alpha, Beta, Radium and Uranium		P,G	5 mL/L HNO <sub>3</sub>		6 m	
Trihalomethanes	G	(vials)	Cool 4°C, Ascorbic Ad	cid	14 d	
Tritium		G	None required			
Radionuclides (not including Iodines and C-14)	)	P,G	5 mL/L HNO3		6 m	
Radon <sup>2</sup>		G	10 mL scintillation cocktail			
TCLP Metals	G	(wide)	None required	180	d (28 d Hg only)	
Volatiles	G	(vials)	Cool 4°C		14 d	
Semi-Volatiles		G	Cool 4°C		7 d extraction; 40 d analysis	

<sup>1</sup> Should only be used in the presence of residual chlorine.

<sup>2</sup> Requires special sampling technique and apparatus.

Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514	Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514
TOTAL PETROLEUM HYDROCARBONS (H,SO,)	OIL & GREASE
Sample ID	Sample ID
Date Time	DateTime
Filtered (.45µ filter) Yes No Sampled By	Filtered (.45µ filter) Yes No Sampled By
Project #	·
Bottle Of	Project # Of
المصادقين والمراكب والمراكب والمراكب	
	·
Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514	Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514
NUTRIENT (H,SO.)	METALS (HNO,)
Sample ID	Sample ID
Date Time	Date Time
Filtered (.45µ filter) YesNo	Filtered (.45µ filter) YesNo
Sampled By	Sampled By
Project #	Project #
Bottle Of	Bottle Of
Accu-Labs Re 4663 Table Mountain Dr. (30)	•
COLIFORM	
	• •
Total - Fecal, Date	·
ChlorinatedNo	•
Water Type: (circle or	:
River-Stream - Lake Sewage-Effluent	- naw Jewaye -
OHAGO-EIIIZOIR	a salla m

# Sample Bottle Labels

Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514	Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514
ORGANICS	SULFIDE (ZnOAc)
Sample ID	Sample ID
DateTime	Sample ID
Filtered (.45µ filter) Yes No	Filtered (.45µ filter) Yes No
Sampled By	Sampled By
Project #	Sampled ByProject #
Bottle Of	Bottle Of
Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650 (303) 277-9514	Accu-Labs Research, Inc. 4663 Table Mountain Dr. Golden, CO 80403-1650. (303) 277-9514
PHENOLS (H,SO.)	General (Non-Preserved)
·	
Sample ID	Sample ID  Date Time
Date Time Filtered (.45µ filter) Yes No	一直:[14 A A A A A B A B A B A A A A A A A A A
Sampled By	Filtered (.45µ filter) Yes No Sampled By Project #
Project #	Project # 100000000000000000000000000000000000
Bottle Of	Bottle Of
<u> </u>	
CYANIDE  Sample IDT  DateT  Filtered (.45µ filter) Yes	Golden, CO 80403-1650 ) 277-9514  (NaOH)  ime
Sampled By	
Project #	

#### ACCU-LABS RESEARCH, INC.

# LABORATORY WORK SHEET DATE: 04/05/91 PAGE 1

2007-37615-2-2

WELL #2

2007-37615-2-1

WELL #1

F 8386 Rev

BARBARA CADE
CADE ENVIRONMENTAL ENGINEERING
3105 ZINNIA CT
GOLDEN, CO 80401

Lab Job Number: 2007-37615-2
Date Samples Received: 03/27/91
Customer PO Number: PROJ# 1QFY91

Telephone: (303) 279-6423

ALR Designation -

Comments -

Sponsor Designation -

2-1L HN03 F 3-1L NON 24 2-1L H2SO4 24 1-1L 0&G 24

Comments

These samples to be disposed of 30 days after the date of this report.

Date Collected -03/27/91 03/27/91 ICP METALS - WA Aluminum - total Barium - total Boron-ICP - total Chromium - total --- N/A ---Iron - total Potassium - total --- N/A ---Silica-ICP - total Silver - total Sodium - total --- N/A ---Zinc - total WATER QUALITY - WA Turbidity WATER ORGANICS - WA Biochemical Oxygen Demand Oil & Grease --- N/A ---NUTRIENTS - WA Phosphorus (as P) ●Total Kjeldahl Nitrogen

#### ACCU-LABS RESEARCH, INC. WATER LABORATORY WORK ORDER

9597 -37617-2

FORM: F 8383 Rev.

DATE PRINTED: 03/27/91

TIME PRINTED: 17:39:53

PAGE: 1

DATE RECEIVED: 03/27/91 PROJECT MANAGER: EH

SUPERVISING DEPARTMENT: WATER LAS

BOTTLE/LOCATION BOTTLE/LOCATION 2-1L TPH 25 2-1L HN03 UF F 2-1L HN03 F F 2-1L NON 25 2-1/2L H2S04 25 2-1/2L NAOH 25

# ICP METALS

PARAMETER	DISSOLVED	TOTAL	REDI AA RM
Barium	All Samples	All Samples	
Boron-ICP	All Samples	All Samples	
Cadmium	All Samples	All Samples	
Calcium	All Samples	All Samples	
Chromium	All Samples	All Samples	
Copper	All Samples	All Samples	<u> </u>
Iron	All Samples	All Samples	
Magnesium	All Samples	All Samples	
Manganese	All Samples	All Samples	
Potassium	All Samples	All Samples	<u> </u>
Silver	All Samples	All Samples	
Sodium	All Samples	All Samples	
Zinc	All Samples	All Samples	
			•
Date complete	ed:	Analyst:	
Date rediges	t completed:	Analyst:	•

# ACCU-LABS RESEARCH, INC. 4663 Table Mountain Drive - Golden, CO 80403 - (303) 277-9514

F 8080 Rev B

# SAMPLE ACKNOWLEDGMENT

TO: BARBARA CADE

CADE ENVIRONMENTAL ENGINEERING

3105 ZINNIA CT

GOLDEN, CO 80401

LAB JOB NUMBER: 2007-37615-2

DATE RECEIVED: 03/27/91 CLIENT P.O. NUMBER: PROJ# 1QFY91

ANTICIPATED COMPLETION DATE:

SAMPLE CONDITION: OK

SAMPLE TYPES RECEIVED:

SAMPLE NUMBERS

WATER

All Samples

ANALYSES TO BE PERFORMED

SAMPLE NUMBERS (See cross reference sheet)

Aluminum (TOTAL)	All	Samples
Barium (TOTAL)		Samples
Boron-ICP (TOTAL)	1	<b>p</b>
Chromium (TOTAL)	2	
Iron (TOTAL)	All	Samples
Potassium (TOTAL)	1	<u> </u>
Silica-ICP (TOTAL)	All	Samples
Silver (TOTAL)		Samples
Sodium (TOTAL)	1	<u>-</u>
Zinc (TOTAL)	Āll	Samples
Turbidity		Samples
Biochemical Oxygen Demand		Samples
Oil & Grease	2	- dp 2,00
Phosphorus (as P)	A11	Samples
Total Kjeldahl Nitrogen		Samples

By	
-	

# ACCU-LABS RESEARCH, INC. 4663 Table Mountain Drive - Golden, CO 80403 - (303) 277-9514

F 8387 Rev A .

#### SAMPLE ACKNOWLEDGMENT

\*\* PAGE 2 \*\*
LAB JOB NUMBER: 2007-37615-2

#### SAMPLE NUMBER CROSS REFERENCE

LAB JOB NUMBER: 2007-37615-2

Lab Sample	Client Sample Name	Date, Time
1	WELL #1	03/27/91,
2	WELL #2	03/27/91,

## Internal Chain-of-Custody

		Į.	ALR Numb	er				
					lumber			
Log-in D	ate			Sa	umple Type			
							•	
· -				·				
Date	Time Out	Analyst	Time In	Analyst	First use of bottle?	Seal Intact?	Analysis Performed	Comments
						:		·
			· · · · · · · · · · · · · · · · · · ·					
			<del></del>				,	
						·		<u> </u>
			<del>-</del>					· · · · · · · · · · · · · · · · · · ·
			·					
<del></del>								· · · · · · · · · · · · · · · · · · ·

Accu-Labs Research,	Inc.	F 8176 Rev. E
Electrode No.	ELECTRODE (	Page of
Analysis:		pH Meter No.:
Procedure No.:	Rev	Date:
Slope:	·	Time:
Avg mv   Concentrat	ion Run 1 Run 2	Analyst:
		D.L.:mg/L
		QC ID:
		QC T.Vmg/L
Sample Number	Dilution Factor	Reading mg/L
Blank		
Spike Blank		
<u> </u>		
•		
Comments:	Ana	lyst checked:
Spike = mL of	mo/L	QC checked:
inmL =mg/L	total Reviewed	and Approved:

Exhibit J-1

Page \_\_\_\_

## VARIANCE FORM

SECTION TO BE COMPLETED BY ANALYST:	;	
ALR No.	Notebook	
Analyst		
Variance from document No.		Rev.
Description of the variance:		
Suspected cause of the variance:		:
SECTION TO BE COMPLETED BY SUPERVISO		
Department: Supervisor:		Date:
Effect of the variance on the data:		
Corrective action (if required):  Verification of corrective action (i	if required):	
Client notification (if required):		•
Client contacted:		Date
ALR staff member initiating contact:		
Comments:		
ALR Variance Number:	_	Date

Exhibit K-1

## QUALITY CONTROL MANUAL CHEMICAL/INDUSTRIAL HYGIENE DIVISION OCTOBER 1989

Chemistry Laboratory

Prepared by: Laboratory Manager

Date: <u>June</u>, 1991

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#### 1.0 PURPOSE

The purpose of this manual is to provide written policies and procedures for testing services provided by the Chemical and Industrial Hygiene Division of Chen-Northern, Inc. These written policies and procedures communicate to all employees the standards of performance expected within our organization. These policies and procedures are consistent with those established by Chen-Northern, Inc. in other areas of practice. Our quality control system covers personnel, equipment, and supervision. Established written guidelines are provided for the development and training of our personnel. Equipment maintenance and calibration schedules are covered.

A quality control coordinator has been designated to monitor the program and report discrepancies to both the Chemical Laboratory Manager and the Area Vice-President.

Our quality control manual serves as a basis for conformance audits by our clients, federal agencies, and certification organizations.

#### 2.0 OBJECTIVES

It is our objective to document the quality of the data generated by our testing services, and thus to maintain a reputation of quality service, including timely, and within budget expectations. We intend to meet these objectives by charging a reasonable fee to our clients and by making a profit for the owners of Chen-Northern, Inc.

Specific objectives of our standards of performance are as follows:

- To develop, review, and update laboratory practices and routine methodologies. This will encompass, among other things, analytical procedures, sampling and sample preparation, and personnel training.
- To monitor and take any corrective action required to maintain a performance level consistent with our guidelines, client needs, and/or regulatory agency requirements.
- To utilize personnel who are trained for the tasks assigned and to provide the supervision and expertise necessary to determine acceptable variations from standard procedures and practices of recognized chemical laboratory techniques.
- To inventory, maintain, and calibrate testing equipment used in our business.
- To review all laboratory data before results are given to the client.
- To participate in inter-laboratory and other round robin evaluation programs to monitor the consistency and level of quality within the chemical laboratory.

#### 3.0 ORGANIZATION

Northern Engineering and Testing, Inc. was founded in 1958 in Great Falls, Montana, serving primarily the Northwest. Major offices were subsequently established in Boise, Idaho; Billings, Montana; Great Falls, Montana; and Salt Lake City, Utah; branch offices in Casper, Wyoming; Pocatello, Idaho; Tri-Cities, Washington; and Evanston, Wyoming serve outlying regions in those areas. Chen and Associates was founded in 1961 in Denver, Colorado serving the Rocky Mountain region. Major offices were subsequently established in Casper, Wyoming; Salt Lake City, Utah; Phoenix, Arizona; and San Antonio, Texas; and branch offices in Colorado Springs and Glenwood Springs, Colorado; Rock Springs and Cheyenne, Wyoming; and Elko, Nevada. In 1987 both firms were acquired by Huntingdon International Holdings (HIH). Chen-Northern, Inc. was founded in 1988 as a result of a merger of Northern Engineering and Testing, Inc. and Chen & Associates, headquartered in Denver, Colorado (see attached organizational chart, Figure 3-1).

The services provided by the chemical laboratory include solid waste analysis, trace metal analysis, air quality monitoring, water and wastewater analysis, mining-related soils analysis and asbestos identification.

As our clients require testing and/or the analysis of materials outside our areas of expertise, we will then employ affiliate laboratories or consultants who have demonstrated expertise and qualifications consistent with the quality-of-service precepts of Chen-Northern.

The organizational structure of the Chemical and Industrial Hygiene Division requires the placement of the quality control function in the laboratory and this is shown in diagram form in Figure 3-2.

The Quality Control Coordinator is responsible for monitoring, recordkeeping, statistical controls and any other function required to maintain the quality assurance system. The Quality Control Coordinator is also responsible for recommending measures to ensure the fulfillment of those objectives of management relating to quality control and to carry out these objectives in an efficient and economical manner.

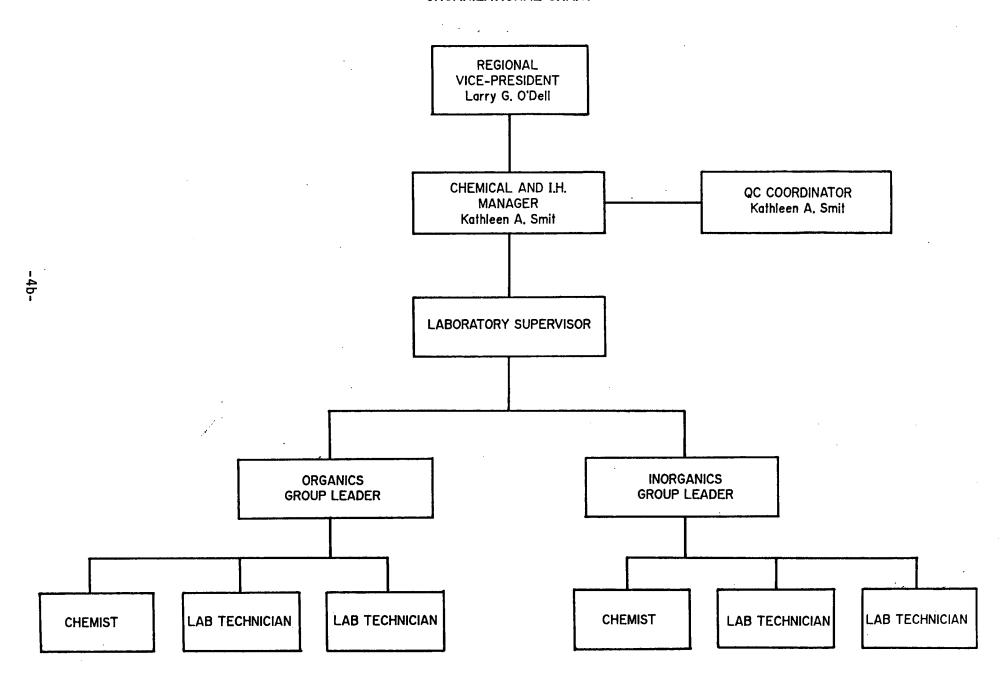
RESERVED

FOR

FUTURE

USE

QUALITY CONTROL LINE AND STAFF ORGANIZATIONAL CHART



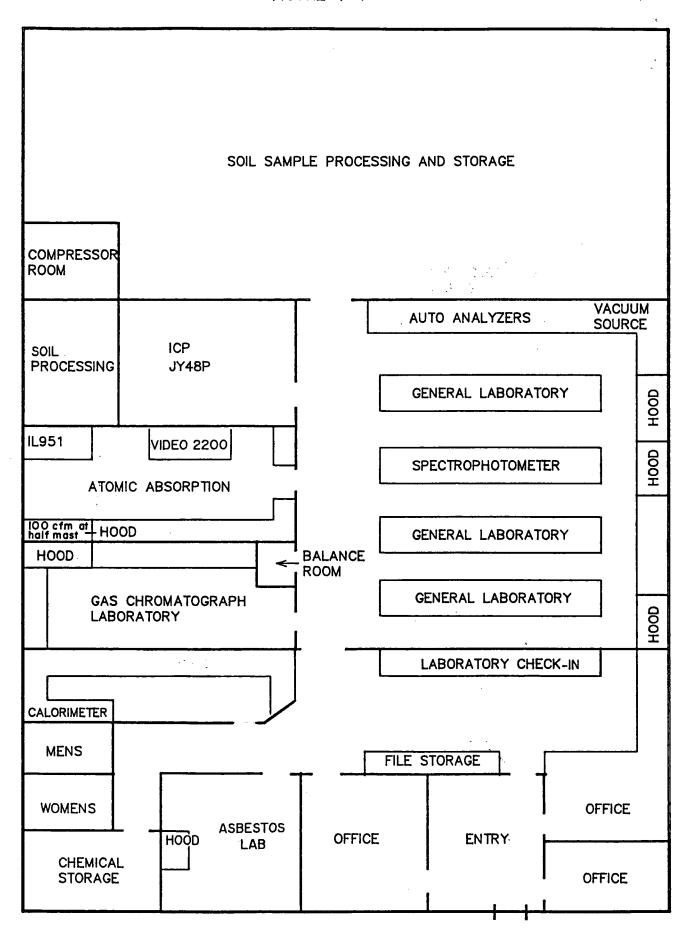
#### 4.0 FACILITIES

The Billings laboratory has a total of 3500 square feet of floor space assigned to laboratory service. The laboratory staff uses the services of the clerical staff that is located in the engineering office. An area of 1500 square feet is used as additional storage area. Figure 4-1 shows the layout of Chen-Northern's facility, including labeled areas for specific laboratory analysis.

The air quality of the laboratory is controlled by the use of ventilation. The laboratory is equipped with five hoods having an air flow of approximately 750 cubic feet per minute to provide ventilation. These hoods are used for exhausting toxic and hazardous fumes and odors. All digestion procedures are completed under the hoods. Their locations are shown in Figure 4-1. There are also canopy hoods for general elimination of hazardous and noxious vapors from the Atomic Absorption and Inductively Coupled Plasma Units.

The laboratory facility is equipped with two forced air heating systems. Air conditioning is also provided by use of two central units.

The noise level in the laboratory is minimized and controlled in order to provide a working environment compatible to the activities associated with wet instrumental and physiochemical methods of analysis. The level of tolerable noise does vary, depending upon the type of activity taking place.



## 5.0 RECORDKEEPING, CHAIN OF CUSTODY, DOCUMENT CONTROL

Records are maintained in the chemical laboratory which identify the sample, request the prescribed analysis, establish the chain of custody, and review and document the report. Records are also maintained on personnel qualifications, equipment status, and quality control reports.

Upon receipt of a sample in the laboratory, it is forwarded to the laboratory manager's representative. The identification information accompanying the sample is recorded on the Sample Log Index (Figure 5-1) and is assigned a number. This is accomplished using the laboratory information management system (LIMS).

A work order (Figure 5-2) and the appropriate tracking charts are completed with the sample identification, laboratory number, and a description of the work requested.

The white copy of the work order is posted in the laboratory with any packing slips, purchase orders, chains of custody or letters of transmittal that accompanied the sample upon delivery. The yellow copy is kept in a time file for scheduling purposes. The pink copy is kept by the business office for invoice verification.

Upon completion of the analyses, the worksheets are checked by a technician and results are entered into the LIMS. The data is summarized in report form (Figure 5-3) by the LIMS and reviewed by the laboratory manager.

The report, accompanying paper work, and work order are compiled by the laboratory manager for publishing and billing. The laboratory manager reviews the published report and accompanying invoice for billing and signs the report for distribution.

The number of requested copies are mailed to the client and the original and the worksheets are placed in the project file. The original report is retained indefinitely and the data worksheets are kept on file three years following the date of the report.

The sample is maintained in the laboratory for seven days after the report has been sent to the client. The sample is either disposed or retained if requested in writing by the client.

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FIGURE 5-1

## SAMPLE LOG SHEET

Lab No.	Client	Date Received	Type of Sample	Tests To Run	Job No.	Billed
		·				·
	· · · · · · · · · · · · · · · · · · ·					
				·		
				,		
		·				



## FIGURE 5-2 CHEM WORK ORDER

Consulting Engineers and Scientists

	Client			<del></del>	Order No	1/12	1.2	<u>'</u> U
	AddressCit	у	· · · · · · · · · · · · · · · · · · ·		Sta	ıte	Zip Co	de
	P.O							
•	Date Received							
LAB NO.	SAMPLE DESCRIPTION		TEST	TS TO B	E PERFORI	/IED	- ' - '	
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			1	++				$\dagger$
			1	<del>                                     </del>		1-1		$\dagger$
								$\dagger$
		DESCRIPTI	NC					
					QUAN	TITY	UNIT PRICE	то
	A	assigned to:			ate Comple			

## FIGURE 5-3

Date Analyzed

Client: Identification: Laboratory Number: Date Sampled:		Date Job No. Sheet
pH, standard units: Conductivity, umhos/cm: Total Dissolved Solids (at 180 C), mg/l: Sodium Adsorption Ratio (SAR):		
CA	TIONS	
Total Hardness as CaCO3: Calcium as Ca: Magnesium as Mg: Sodium as Na: Potassium as K:	mg/l mg/l mg/l mg/l mg/l Total Cations:	meq/l meq/l meq/l meq/l meq/l meq/l
A	NIONS	•
Total Alkalinity as CaCO3: Bicarbonate Alkalinity as HCO3 Carbonate Alkalinity as CO3: Hydroxide Alkalinity as OH: Chloride as Cl: Fluoride as F: Nitrate + Nitrite as N: Sulfate as SO4:  Cation-A	mg/l mg/l mg/l mg/l mg/l mg/l mg/l Total Anions: nion Difference:	meq/l meq/l meq/l meq/l meq/l meq/l meq/l meq/l meq/l
Ortho-Phosphate as P:	mg/l	
TRACE ELEMEN	NTS, DISSOLVED	
Aluminum as Al: Boron as B: Cadmium as Cd: Copper as Cu: Iron as Fe: Lead as Pb: Manganese as Mn: Mercury as Hg: Selenium as Se: Silver as Ag: Vanadium as V: Zinc as Zn:	mg/l mg/l mg/l mg/l mg/l mg/l mg/l mg/l	

## 6.0 QUALITY CONTROL POLICIES

Policies which are implemented by the chemical laboratory to achieve and support the quality objectives are listed in this section. Details for implementing these policies appear in later sections of this manual.

Administrative and technical review of laboratory reports and records to assure validity and uniformity is provided by the assigned QA coordinator, the technician generating the data and the laboratory manager.

Complete and current analytical methods and procedures and analytical instrument operating instructions are made available to laboratory personnel. Data and calibration records are updated upon their generation by laboratory personnel.

Only skilled and trained personnel are employed. The equipment used in the laboratory is maintained and calibrated on a regular basis as described by the method used for quantification of the analyte of interest. All chemicals, reagents, and precision equipment used in the laboratory are certified or standardized and documented on a regular basis.

The following flow diagram (Figure 6-1) indicates the quality control routine used by Chen-Northern. This flow diagram governs the actions of the technicians as they analyze samples for any parameter. A generalized rule for all tests includes the following salient points:

Standards traceable to National Bureau of Standards or primary standards as
defined by the American Chemical Society and at least one blank (reagent
blank) shall be run with any number of samples to be analyzed. In the event

more than 20 samples are analyzed in one batch, the standards shall be verified after each set of 20 analyses. Internal standards are not currently used in our operation.

- 2. Each inorganic batch shall include 10 percent duplicate analyses, 10 percent spike analyses and at least one standard reference material. Reference material concentration recoveries shall be compared to accuracy acceptance criteria. Reference material concentrations shall be plotted on control charts for identification of bias.
- 3. Each organic batch shall include at least one daily spike and 10% duplicate analysis. Appropriate blank analyses will be accomplished as needed. For volatile organics, the recovery of a surrogate material will be evaluated for each purged sample.
- 4. All raw data such as tare weights, titration volumes, absorbances, peak heights, fiber counts, etc. will be recorded in the laboratory notebook. Correlation coefficients, slopes and y intercepts shall be recorded for Beer's law curves used for data generation.
- 5. Dates and technician initials shall be recorded with raw data. Notes of unusual circumstances or special sample treatments shall be entered in the lab notebook.
- 6. Raw data shall be converted to concentration units by using the calculations given in the appropriate written method. Deviations from this shall be recorded.

In an effort to verify control and demonstrate capability as an analytical laboratory, Chen-Northern subscribes to the following quality audit programs:

- EPA SDWA Drinking Water Supply Audit Biannually
- EPA NPDES Wastewater Source Audit Biannually
- NIOSH PAT Program Asbestos Quarterly
- NBS Asbestos Identification Audit Biannually
- Utah State University Soil Analysis Round Robin Annually

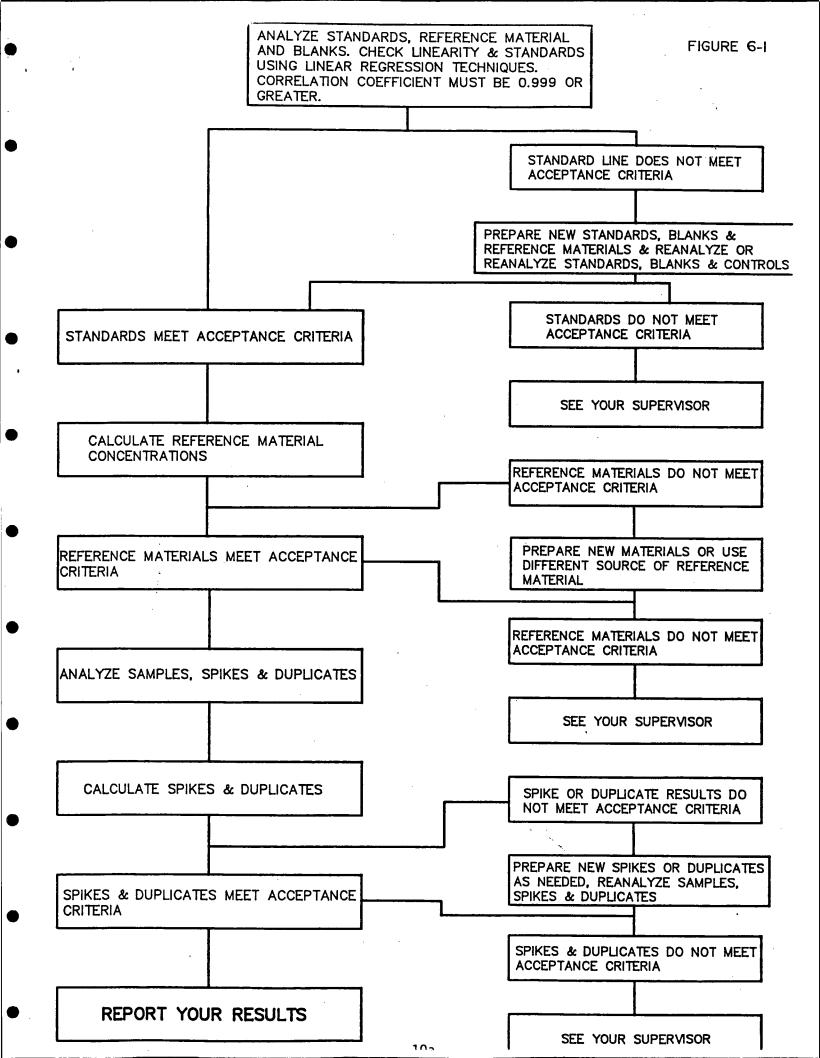
Quality control acceptance criteria for precision and accuracy is generated using data produced in our laboratory. For precision, the acceptable range of duplicate concentrations is determined by calculation of critical differences of data produced in the past. Using the following equation, a critical difference or "Range of duplicate" is established for each range of concentration of any particular analyte.

Critical Difference = 
$$(X_1 - X_2) \times 3.27$$

For accuracy, the upper and lower confidence levels are calculated by using the following equation for each range of concentration for any particular analyte.

Blind field standards will be analyzed on a project by project basis. Reports of performance on blind field standards will be made to the project file. Any discrepancies (data outliers) shall be identified and the source of error eliminated.

Reports of quality assurance/quality control efforts are made verbally to the management of Chen-Northern upon request. A schedule of these reports has not been devised.



#### **DEFINITION OF TERMS:**

Range:

The range is the boundary of lowest to highest values to

which a statement pertains.

Rc:

Critical range; the maximum allowable difference

between a sample value and its duplicate value.

LCL:

Lower control limit; the smallest acceptable percent

recovery allowed for a spiked sample.

UCL:

Upper control limit; the highest acceptable percent

recovery allowed for a spiked sample.

Example:

A sample is analyzed for calcium. Values determined were 33.4 and 39.6. Is

this within acceptable duplicate criteria?

From the quality control acceptance criteria (Table 1) for the range of 10-50, the maximum allowable difference in calcium duplicate is 5. Since the difference of these values is 6.2, the sample determinations are not acceptable.

Example:

A sample is analyzed for dissolved arsenic content. This value is 0.011 ppm. The sample is spiked to a total concentration of 0.020 ppm and 0.017 ppm is recovered. Is this acceptable?

In the range 0.010 to 0.050, the lowest percent recovery allowed is 90 percent (on a dissolved determination); the highest percent recovery allowed is 109 percent.

0.017 x 100 is 85 percent recovery and0.020 determination does not meet with the acceptance criteria of the quality control acceptance criteria.

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	Precision	Precision		Accuracy		
Parameter	Range Of R	ange Of uplicate mg/l	LCL % Recovery	UCL % Recovery	Detection Limit, mg/l	
Acidity	1.0 - 10.0	2.8	0	0	1.0	
•	10.0 - 100.0	5.9	0	0		
	100.0 - 500.0	20	0	0		
	500.0 - 1000.0	40	0	0 0 0		
	1000.0 - 10000.0	50	0	0		
Alkalinity	1.0 - 50.0	3	93	105	1.0	
J	50.0 - 100.0	10	93	105		
	100.0 - 500.0	13	93	105		
	500.0 - 1000.0	35	93	105		
Aluminum	0.1 - 1.0	0.2	89	110	0.1	
	1.0 - 5.0	0.3	89	110		
	5.0 - 10.00	0.6	89	110		
Ammonia as N	0.05 - 0.20	0.05	87	111		
	0.2 - 1.0	0.07	87	111	0.05	
	1.0 - 10.0	0.6	87	111		
Antimony	0.05 - 0.50	0.05	85	108	0.05	
· · · ···- ··•	0.50 - 5.00	0.10	85	108		
	5.0 - 50.0	0.50	85	108		
Arsenic	0.002 - 0.010	0.002	86	112		
J <del>.</del>	0.010 - 0.050		88	110	0.002	
	0.050 - 0.100		89	110		

<sup>\*</sup> May be difficult to achieve, use other quality indicators as an aid in data evaluation.

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Parameter	Range Of Concentration mg/l	Range Of Duplicate	Accu LCL Recovery	UCL % Recovery	Detection Limit, mg/l
Barium	0.1 - 1.0 1.0 - 10.0	0.1 0.3	93 93	105 105	0.1
Benzene	A11 20	% of Average Value	80	120	0.001
Beryllium	0.005 - 0.05 0.050 - 0.10		84 84	105 105	0.005
BOD	1.0 - 10.0 10.0 - 25.0 25.0 - 100.0	3 9 15	0 0 0	0 0 0	1.0
Boron	0.1 - 1.0 1.0 - 5.0 5.0 - 10.0 10.0 - 100.0	0.1 0.4 1.0 6	89 89 89 89	109 109 100 109	0.1
Cadmium	0.0001 - 0.005 0.005 - 0.010 0.010 - 0.050 0.050 - 0.100	0.0004 0.004 0.009 0.015	81 88 88 88	118 105 105 105	0.0001 0.005
Calcium	0.1 - 2.0 2.0 - 10.0 10.0 - 50.0 50.0 - 100.0 100.0 - 200.0 200.0 - 500.0 500.0 - 1000.0	0.3 (Tentative 0.5 (Tentative 4 7 10 20		110 110 105 105 105 105	0.1 0.1

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	Precisio				
Parameter		Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	Detection Limit, mg/l
COD	1.0 - 10.0 10.0 - 100.0 100.0 - 500.0	2 12 21	71 69 70	105 105 105	1.0
Chloride	1.0 - 10.0 10.0 - 50.0 50.0 - 100.0 100.0 - 1000.0	1 3.0 6.0 16	86 94 94 94	111 105 105 105	1.0
Chromium	0.002 - 0.02 0.02 - 0.20 0.20 - 1.00 1.00 - 10.00	0.004 0.02 0.10 0.20	90 91 94 94	121 107 108 110	0.002 0.02
Conductivity	0.1 - 1.0 1.0 - 100.0 100.0 - 1000.0 1000.0 - 5000.0 5000.0 - 10000.	160	0 0 0 0	0 0 0 0	0.1
Copper	0.001 - 0,02 0.02 - 0.20 0.20 - 0.50 0.5 - 2.00 2.00 - 10.00	0.004 0.02 0.04 0.10 0.2	92 89 91 90	116 116 109 107	0.01 0.02

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# QUALITY CONTROL ACCEPTANCE CRITERIA PRECISION & ACCURACY REPORT

-----Accuracy----------Precision-----Range Of Range Of Concentration Duplicate LCL UCL Detection Parameter mg/1mg/1% Recovery % Recovery Limit, mg/1 Ethylbenzene A11 ± 20% of Average 80 120 0.001 Fluoride 0.05 - 0.200.04 84 108 0.05 0.2 - 1.0 0.07 94 103 1.0 - 2.0 0.1 93 106 93 2.0 - 5.00.3 112 5.0 - 50.01.3 93 112 Iron 92 0.05 - 0.50.07 106 0.05 0.50 - 1.00.1 92 106 0.001 1.00 - 5.0 0.20 92 106 5.00 - 10.00.32 92 106 10.00 - 50.0 1.0 92 106 Lead 0.001 - 0.0200.002 81 118 0.02 - 0.1082 0.03 0.02 109 0.10 - 1.00 0.08 89 105 1.00 - 10.000.16 105 Magnesium 0.1 - 2.00.3 (Tentative) 89 110 2.0 - 10.093 102 10.0 - 50.0 93 102 0.1 50.0 - 100.0 93 103 100.0 - 500.0 10 94 102 500.0 - 1000.0 50 93 102 Manganese 0.02 - 0.100.03 87 0.02 107 0.10 - 0.500.03 92 107 0.50 - 1.0090 0.05 108 1.00 - 5.00 0.28 95 107 5.00 - 10.00 0.50 90 107 10.00 - 50.00 1.0 90 107

May be difficult to achieve, use precision guidelines to evaluate out of range spikes.

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	Accuracy					
Parameter	Range Of	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	Detection Limit, mg/l	
Mercury	0.0002 - 0.0020 0.0020 - 0.006 0.0060 - 0.020	0.0003 0.0005 0.0010	84 84 84	110 110 110	0.0002	
Molybdenum	0.05 - 0.100 0.10 - 1.00	0.02 0.08	91 92	104 105	0.05	
Nickel	0.02 - 0.10 0.10 - 1.00	0.02 0.05	86 88	102 102	0.02	
Nitrate as N	0.01 - 0.10 0.10 - 0.50 0.50 - 2.00 2.0 - 10.0	0.02 0.04 0.10 0.3	91 90 90	109 106 106	0.01	
Petroleum Hydrocarbons	All 20% of	Average Value	0	120	Varies	
рН	0.10 - 14.0	0.16	0	0	0.10	
Phosphate as P	0.005 - 0.020 0.020 - 0.10 0.10 - 0.50 0.50 - 1.00	0.005 0.02 0.09 0.09	84 84 93 93	112 112 108 108	0.005	
Potassium	0.5 - 2.00 2.00 - 10.0 10.0 - 50.0	0.2 0.5 3	90 94 94	105 105 104	0.5	
Selenium  * May be difficul	0.002 - 0.010 0.010 - 0.050 0.050 - 0.100 t to achieve, use	0.002 0.004 0.010 other quality in	*89 89 88 ndicators as an a	*110 110 110 id in data evaluation.	0.002	

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- * •	Precision		Accuracy		
Parameter		nge Of plicate mg/l	LCL % Recovery	UCL % Recovery	Detection Limit, mg/l
Silver	0.0001 - 0.0050	0.0002	88	118	0.0001
	0.0050 - 0.010	0.0006	88	118	0.02
	0.01 - 0.10	0.02	85	115	
	0.10 - 1.00	0.08	90	108	
Sodium (Tentative	e) 0.1 - 2.0	0.3	89	110	•
(1011040111	2.0 - 10.0		90	105	0.1
	10.0 - 100.0	2 5	90	105	
	100.0 - 500.0	15	91	105	
	500.0 - 1000.0	50	91	105	
Sulfate	1.0 - 50.0	6	89	111	1.0
Jarrate	50.0 - 100.0	12	92	109	
	100.0 - 500.0	33	91	109	
	500.0 - 1000.0	63	91	109	
	1000.0 - 5000.0	90	93	110	
Dissolved	1.0 - 50.0	5	0	0	
Solids	50.0 - 200.0	16	0	0	1.0
	200.0 - 500.0	20	0	0	
	500.0 - 1000.0	38	0	0	
	1000.0 - 5000.0	129	0	0	
	5000.0 - 10000.0	375	0	0	
Suspended	1.0 - 10.0	2	0	0	1.0
Solids	10.0 - 50.0	7	0	0	
	50.0 - 100.0	14	0	0	
	100.0 - 500.0	25	0	0	• .
	500.0 - 1000.0	50	0	0	
	1000.0 - 10000.0	95% of Aver	rage value		
Toluene	A11 20% o	f Average Val	lue 80	120	0.001

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	Pre	cision	Accuracy		
Parameter	Range Of Concentration mg/l	Range Of	LCL % Recovery	UCL % Recovery	Detection Limit, mg/l
Vanadium	0.20 -	1.0 0.1	83	101	0.2
Volatile Organics	All	20% of Average Value	80	120	Varies
Xylenes	A11	20% of Average Value	80	120	0.001
Zinc	0.02 - 0.10 - 0.5 - 2.00 -	0.5 0.004 2.0 0.10	88 90 90 90	105 105 105 105	0.02

## 7.0 CALIBRATION AND PREVENTIVE MAINTENANCE

Test instruments are calibrated on regular intervals recommended by the manufacturer and as required by ASTM, EPA or other methods. Calibration of all equipment used and documentation of the calibration will be performed by individuals assigned by the laboratory manager or by an independent calibration firm.

Equipment calibrated includes any equipment to be used for analytical testing. The standards, whether reagent or apparatus, used for calibration of equipment are calibrated against a standard traceable to NBS or other recognized physical or chemical constants.

Calibration procedures used are specified by the manufacturer, regulatory agencies, or method. The procedure provides the specific instructions in a step-by-step detail for obtaining and documenting the results. The data are kept on file in the chemical laboratory with the data generated using that instrument calibration. A chronological list of calibration due dates is maintained in a central filing system to maintain proper calibration intervals.

## 8.0 PROCUREMENT CONTROL AND REAGENT QUALITY

Materials, reagents, and chemicals are received, labeled, stored and issued at Chen-Northern's chemical laboratory according to technical and quality requirements.

Quality standards apply to the vendor supplying the laboratory with analytical materials. Containers, materials or reagents and their packing slips must be marked with the vendor's name and address, the name of the material, the vendor's lot number, quantity and expiration date. Materials received must meet the most recent specifications and the materials must be properly identified.

Purchase orders, packing list certifications, and receiving papers are retained in a central file and are used as a quality control check on the materials received.

The control of incoming materials and reagents is handled by the individual who placed the order. Upon receipt of the supplies, the ordering information is compared with the receiving information. If a discrepancy is found that may affect the quality of the product, the materials are returned. If accepted, the individual ordering the material places the date on the label of the reagent or material, along with the date when the material is no longer capable of peak performance (shelf life). The laboratory manager determines the shelf life or, for certain chemicals, a shelf life is provided by the manufacturer. A first-in first-out usage is maintained by the users. The laboratory manager surveys the store room monthly and disposes of any materials, chemicals or reagents approaching the shelf life expiration date and reorders the chemical.

All organic and inorganic chemicals used in the laboratory for the preparation of primary standards and other solutions conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society. If other grades are used, it first must be ascertained that the chemical is of sufficiently high

purity to permit its use without lessening the accuracy of the analysis. Reagents and solvents are stored in borosilicate glass bottles, metal or polyethylene containers, whichever is appropriate according to method specifications.

Reagents and solvents sensitive to light or temperature are stored in dark bottles or in a cool dark place. The concentration and composition of many reagents are susceptible to change over a short period of time. All reagents and standards are labeled and dated when prepared to monitor their shelf life. The estimated shelf life for the common reagents used are shown in Table 8-1.

Control or reference samples are analyzed with each set of samples for all analytical procedures to insure that the reagents used have not degraded or become contaminated and the instrument performance is meeting its specifications.

Gases used in the laboratory can be classified to serve one of three functions: fuel, oxidant or carrier.

The following is a list of the types of gases used:

<u>Type</u>	<u>Parameter</u>	Use (function)
Air, zero grade Acetylene, Commercial	Organic Analysis	Oxidant
Grade	Metals Analysis	Fuel
Nitrous Oxide	Metals Analysis	Oxidant
Compressor Air -	•	
Supplied by Compressor	Metals Analysis	Oxidant
Argon	Metals Analysis	Carrier
Nitrogen	Metals Analysis	Carrier
Helium, ultra purity	Organics Analysis	Carrier
Hydrogen, ultra purity	Organics Analysis	Fuel

Most fuels and oxidant gases used for atomic absorption work are of commercial grade. Air supplied by a compressor is passed through a filter to remove any oil, water and trace metals from the line for metals analysis only.

There are three grades of water used in the laboratory. Tap Water; Type II ASTM Reagent Water; and Type I ASTM Reagent Water. A description of the various waters used follows:

- 1. Tap Water The tap water used in the laboratory is from the Billings City water supply. Its primary use is for the washing of glassware and containers.
- 2. Type II ASTM Reagent Water This water is produced by passing tap water through a Millipore reverse osmosis system and Barnstead polishing cartridges. This water has a greater than 1 megaohm resistance. This water is primarily used for preparing reagents, solutions and standards. It is also used as the final rinse for most of the laboratory glassware and containers.
- 3. Type I ASTM Reagent Water This higher quality water is produced by treating the Type II water with a Millipore Super Q four bed recirculating system. This water is available on demand at up to 3 gpm.

The water is checked to determine if each type is meeting the criteria listed in Table 8-2. The resistance is monitored weekly at various laboratory outlets to verify the quality of distilled water.

The type of glassware and its cleanliness are important factors in obtaining accurate analytical results in the laboratory. The glassware used in the laboratory for volumetric and colorimetric analyses are borosilicate Kimax or Pyrex brand glass.

Other glassware meets the Class A specifications of the National Bureau of Standards with reference to capacity and delivery.

The cleaning method for the glassware is dependent upon the substances that are to be removed and the use of the glassware. Water-soluble substances are removed with tap water and demineralized or distilled water. Other substances more difficult to remove require the use of an acid cleaning solution as well. Special cleaning techniques are necessary for glassware used for certain laboratory analysis.

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#### TABLE 8-1

#### REAGENT SHELF LIFE LIST

<u>Parameter</u>	Reagent	Container <u>Type</u>	Storage or Shelf Life	<u>Remarks</u>
Asbestos Fibers	Mounting Solution	Balsam Bottle	3 Months	None
Asbestos Identifi- cation	Dispersion Liquids	Amber Glass	1 Year	None
Ammonia Ammonia Ammonia	Stock Ammonia Mixed Indicator Nessler Reagent	Glass Glass Dark Glass with	1 Month 3 Months 6 Months	
Ammonia Ammonia Ammonia Ammonia	Borate Buffer Solution Sodium Hydroxide Boric Acid Solution H <sub>2</sub> SO <sub>4</sub> 0.02 N	rubber stopper Glass or Plastic Plastic Glass Glass or Plastic	3 Months 3 Months 1 Month 3 Months	
No.	- 1	Plastic	standardize  3 Months	daily
Boron  Bromide Bromide Bromide Bromide	Stock Boron Solution  Acetate Buffer Solution Chloromine-T Solution Phenol Red Indicator Sodium Thiosulfate	Glass Brown Glass Glass or Plastic Glass or Plastic standardize daily	3 Months 3 Months 3 Months 3 Months 3 Months	Store at 4°C
Bromide	Stock Bromide Solution	Glass or Plastic	3 Months	
Chlorides Chlorides Chlorides	Standard Sodium Chloride Nitric Acid lN Sodium Hydroxide lN	Plastic Glass or Plastic Plastic	3 Months 3 Months 3 Months	
Low Chlorides Low Chlorides	Indicator Reagent Standard Mercuric Nitrate	Brown Glass Brown Glass	1 Month 3 Months standardize	Store at 4°C daily

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### TABLE 8-1 (cont.)

<u>Parameter</u>	Reagent	Container Type	Storage or <u>Shelf Life</u>	<u>Remarks</u>
High Chlorides High Chlorides	Mixed Indicator Reagent Strong Standard Mercuir	Glass	3 Months 3 Months -	
might office racs	Nitrate	Brown Glass	standardize daily	<b>/</b>
Chlorine Residual				
Iodometric	Standard Sodium Thiosulfate	Glass or Plastic	3 Months	Standardize daily
Iodometric	Standard Iodine Solution	All Glass Dark	3 Months	Standardize daily
Iodometric	Starch Solution	Glass or Plastic	3 Months	•
Chromium, Hex	APDC Solution	Amber Glass	3 Months or	
			Discoloration	
Cyanide	Sodium Hydroxide			
	Solution	Plastic	3 Months	
Cyanide	Cuprous Chloride Reagent	Glass or Plastic		
	· · · · · · · · · · · · · · · · · · ·	with Copper Wire in Bottle		
Cyanide	Sodium Dihydrogen Phosphate	Plastic	3 Months S	Store at 4°C
Cyanide	Stock Cyanide Solution	Glass or Plastic	3 Months	
Cyanide	Standard Silver Nitrate	Glass	3 Months	
Cyanide	Rhodanine Indicator	Glass	3 Months	
Cyanide	Chloramine T	Glass or Plastic		Store at 4°C
Cyanide	Pyridine Bartituric Acid	Dark Glass	1 Month	
Cyanide	Stock Cyanide Solution	Plastic or Glass	6 Months	
Fluoride	Standard Fluoride Solution	Glass	3 Months	
Fluoride	Stock Fluoride Solution	Glass	3 Months	
Fluoride	TISAB Reagent	Glass	1 Month	
Hardness	Buffer Solution	Plastic or Pyrex Glass		NOTE: Be sure to have bottle stop- pered tightly to
				prevent loss of HN <sub>3</sub>

# Chen-Northern, Inc.

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TABLE 8-1 (cont.)

<u>Parameter</u>	Reagent	Container Type	Storage or <u>Shelf Life</u>	<u>Remarks</u>
Hardness Hardness	EDTA Titrant 0.01M Standard Calcium Solution	Plastic Glass or Plastic	3 Months 3 Months	Standardize daily
Nitrogen -				
Kjeldahl & Ammonia	Alkaline Phenol	Plastic or Glass	3 Months	
Kjeldahl & Ammonia	Sodium Hypochlorite	Plastic	1 Month	
Kjeldahl & Ammonia	Disgestant Mixture	Glass	3 Months	
Kjeldahl & Ammonia	Stock Ammonium Chloride	Glass or Plastic	3 Months	
Kjeldahl & Ammonia	Digestion Reagent	Glass	3 Months	
Kjeldahl & Ammonia	Sodium Hydroxide/Thiosulfate	Plastic	3 Months	
Kjeldahl & Ammonia	Sodium Thiosulfate	Glass	1 Month	
, Kjeldahl & Ammonia	Mix Indicator	Glass Stoppered	1 Month	
Kjeldahl & Ammonia	Indicator Boric Acid	Glass	1 Month	
Kjeldahl & Ammonia	0.02 N Sulfuric Acid	Glass	3 Months	
Nitrogen -				
Nitrate-Nitrite	Combined Color Reagent	Glass	3 Months	
Nitrate-Nitrite	Ammonium Chloride	Plastic or Glass	3 Months	
Nitrate-Nitrite	Stock Nitrate Solution	Plastic or Glass	3 Months	
Nitrate-Nitrite	Standard Copper Sulfate	Glass	3 Months	
Nitrate-Nitrite	EDTA	Glass or Plastic	3 Months	
Nitrate-Nitrite	EDTA	Glass or Plastic	3 Months	
Nitrate-Nitrite	Sulfanilic Acid Reagent	Glass or Plastic	3 Months	
Nitrate-Nitrite	Naphthylamińe Reagent	Glass	3 Months	
Nitrate-Nitrite	Sodium Acetate Buffer			
	Solution	Glass or Plastic	3 Months	
Nitrate-Nitrite	Stock Nitrite Solution	Glass or Plastic	1 Month	
Petroleum Hydrocarbons	Standard Solution	Glass	4 Weeks	

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TABLE 8-1 (cont.)

<u>Parameter</u>	<u>Reagent</u>	Container <u>Type</u>	Storage or <u>Shelf Life</u>	<u>Remarks</u>
Pheno1	Aminoantipyrine and			
	Potassium Ferricyanide	Glass	1 Day	
Phenol	Ammonium Chloride	Glass or Plastic	3 Months	
Pheno1	Ammonium Hydroxide	Glass	3 Months	
Pheno1	Bromate Bromide Solution	Glass	3 Months	
Pheno1	Starch Solution	Glass or Plastic	3 Months	
Pheno1	Potassium Ferricyanide	Glass or Plastic	1 Week	
Pheno1	Stock Phenol Solution	Glass	1 Month	
Phosphate -				
Ascorbic Acid	5N Sulfuric Acid	Glass	3 Months	
Ascorbic Acid	Antimony Potassium Tartrate	Dark Glass and Stoppers	3 Months	Store at 4°C
& Ascorbic Acid	Ammonium Molybdate	Plastic	3 Months	Store at 4°C
Ascorbic Acid	Ascorbic Acid	Glass	1 Week	Store at 4°C
Ascorbic Acid	11 N Sulfuric Acid	Glass	3 Months	
Ascorbic Acid	Stock Phosphate	Glass	3 Months	Store at 4°C
Silica	Sulfuric Acid 1N	Plastic	3 Months	
Silica	Hydrochloric Acid 1+1	Plastic	3 Months	
Silica	Ammonium Molybdate Reagent	Plastic	3 Months	
Silica	Oxalic Acid	Plastic	3 Months	
Silica	Stock Silica Solution	Plastic	3 Months	
Sulfate	Barium Chloride 10% Reagent	Glass or Plastic	6 Months	
	Standard Sulfate Solution	Glass or Plastic	3 Months	

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TABLE 8-1 (cont.)

<u>Parameter</u>	Reagent	Container <u>Type</u>	Storage or <u>Shelf Life</u>	<u>Remarks</u>
Sulfide	Hydrochloric Acid 6N	Glass	3 Months	
Sulfide	Standard Iodine Solution	Glass	3 Months	
Sulfide	Standard Sodium Thiosulfate	Glass or Plastic	3 Months	Standardize daily
Sulfide	Starch Solution	Glass or Plastic	3 Months	Standardize daily
Sulfite	Sulfuric Acid 1+1	Glass or Plastic	3 Months	•
Sulfite	Starch Indicator	Glass or Plastic	3 Months	
Sulfite	Standard Potassium Iodide-Iodate	Glass or Plastic	3 Months	Standardize daily
Sulfur	Methyl Orange Rinse Solution	Glass or Plastic	3 Months	
Sulfur	Sodium Carbonate Titrant	Glass or Plastic	3 Months	
Sulfur	Ammonium Hydroxide	Glass or Plastic	3 Months	
Sulfur	Bromine Water	Glass or Plastic	3 Months	
Sulfur	5 N Sodium Hydroxide	Plastic	3 Months	
Sulfur	3 N Hydrochloric Acid	Glass or Plastic	3 Months	
Sulfur	10% Barium Chloride Solution	Glass or Plastic	3 Months	
Volatile Organics	Standard Solution	Glass	4 Weeks	

# TABLE 8-2 WATER QUALITY CRITERIA

	Type I	Type II
Total matter, max. mg/litre	0.1	0.1
Electrical conductivity, max. umho/cm at 298 K (25°C)	0.06	1.0
Electrical resistivity, min. M cm at 298 K (25°C)	16.67	1.0
pH at 298 K (25°C)	Α	Ą
Minimum color retention time of potassium permanganate, minutes	60	60
,	00	00
Maximum soluble silica	not detectable	not detectable

#### Microbiological classification $^{B}$

 $<sup>^{\</sup>rm B}{\rm When}$  bacterial levels need to be controlled, reagent grade types should be further classified as follows:

	Type A	Type B	Type C
Maximum total bacteria count	0/m1	10/ml	100/ml

<sup>&</sup>lt;sup>A</sup>The measurement of pH in Type I and II reagent waters is meaningless and has been eliminated from the procedure, since electrodes used in this test contaminate the water.

#### 9.0 TRAINING

All Chen-Northern employees are trained to perform the tasks assigned to them. This consists of on-the-job training. Short courses and specialty conferences are included when appropriate. All technical personnel are required to attend an indoctrination program administered by the laboratory manager. The indoctrination program covers employment requirements, policies, procedures, and objectives of Chen-Northern and stresses the aspects of its quality assurance program.

All new employees are considered probationary for a period of six months from the time of employment. The training program for new personnel is administered by the laboratory manager and consists of on-the-job training directed by the laboratory supervisor. The trainee is required to read the appropriate standards, methods or standard operating procedures and to become familiar with the equipment and measurements used in testing. The trainee observes an experienced operator perform the tests and then performs the test under the direct supervision of the operator.

Before any test is performed by a trainee without direct supervision, the laboratory supervisor observes the trainee performing the test and then initials the trainee's "Personnel Proficiency Check Sheet" (Figure 9-1). Maintenance of the proficiency check sheet system is the responsibility of the laboratory manager. The personnel progress check sheets are filed in the chemical laboratory. The progress check sheets are examined annually to determine employee growth, supervisory efficiency, compatibility of job objectives and overall job knowledge, including a review of wage/salary to determine if compensation is compatible with job progress, duties and responsibilities.

It is Chen-Northern's policy to provide for the continuing training and development of its technical personnel. The program is administered by the laboratory manager and provides for the following:

- a) Selected external programs for attendance by key personnel. Program selection and attendance must have approval of both the laboratory manager and the Regional Vice-President.
- b) Selected special training for specialized positions or for newly created positions may include assistance for attendance at accredited educational institutions. The laboratory manager has the responsibility for selecting and recommending participation in these programs.
- c) The laboratory manager will review and evaluate the development programs once a year regarding the effectiveness of the programs and changes to be made.

#### FIGURE 9-1

## PERSONNEL PROFICIENCY CHECK SHEET

Employee Name:		Date of I	Date of Employment:		
Analyses	Equipment Operation	Date	Initial of Lab Manager		
,					
Record of C	Continuing Education:				
Seminars, C	lasses, etc.	<u>Da</u>	te <u>CEU's</u>		

#### 10. SAFETY

Appropriate safety techniques and procedures are required with the continual expansion and sophistication of techniques, chemicals and equipment used in an environmental laboratory. It is never assumed that personnel at any level of work have adequate information about laboratory safety. For this reason, the need for a training program is recognized to insure a safe laboratory environment.

This program involves the availability of proper safety equipment and adequate personnel training. The following is a list of the safety equipment located in the laboratory:

- Emergency shower
- Gas mask
- Eye wash fountain
- Eye wash solution
- Fire extinguishers
- Emergency blanket
- First aid kit
- Thermal gloves
- Safety glasses
- Laboratory coats
- Fume exhaust hoods

Fume hoods are provided in the laboratory for safe use of gaseous or toxic reagents. The reagents are stored in proper containers at ambient temperature in specifically designated areas of the laboratory. These areas are segregated to avoid contact of incompatible hazardous materials. Proper grounding of electrical equipment in the laboratory is inspected and monitored as a part of the routine preventative maintenance program.

#### 11.0 ANALYTICAL METHODS

The analytical methods used by Chen-Northern for the analysis of water and wastes have been documented by the U.S. Environmental Protection Agency as approved methodologies, under the National Pollutant Discharge Elimination System (NPDES) Permit Program or the Safe Drinking Water Act. Most of the procedures used by Chen-Northern are from the following sources: Standard Methods for the Examination of Water and Wastewater; Methods for Chemical Analysis of Water and Wastes, U.S. EPA; ASTM Water, EPA SW-846, "Test Method for Evaluating Solid Wastes."

Methods for the analysis of materials for asbestos are from NIOSH Manual of Analytical Methods and EPA's Interim Method for the Determination of Asbestos in Bulk Insulation Samples, EPA 600/4-82-020. Methods used for the analysis of soils and overburden are from the USDA Handbook 60, Diagnosis and Improvement of Saline and Alkali Soils and American Society of Agronomy Monograph 9, Methods of Soil Analysis and others.

Methodologies employed are documented in Table 11-1 for specific analyses. These methods specify at a minimum:

- appropriate instrumentation and equipment
- instrument calibration
- reagent quality and concentration
- reagent standardization
- analytical procedure
- method of calculation of results

These methods generally describe the conversion of raw data (absorbances, weights, volumes, etc.) to concentration units. As a matter of standard practice, concentrations as a result of gravimetric analysis are determined by calculation using weights and

volumes measured in the test. Concentrations, as a result of spectrophotometric analysis are calculated by comparing absorbance units of standards and samples using linear regression equations.

TABLE 11-1
ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Acidity	Titrimetric, EPA Method 305.1
Alkalinity	Titrimetric, EPA Method 310.1 Colormetric, Automated Methyl Orange, EPA Method 310.2
Aluminum	Direct Aspiration, ICP AA, Furnace, EPA Method 202.2
Antimony	Direct Aspiration, ICP Furnace, AA, EPA Method 204.1
Arsenic	Hydride, AA, EPA 206.3 Furnace, AA, EPA 206.2
Barium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 208.1
Beryllium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 210.1 Furnace, AA, EPA Method 208.2
Benzene	Gas Chromatograph EPA 602, 8020
Boron	Direct Aspiration, ICP
Cadmium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 213.1 Furnace, AA, EPA Method 213.2
Calcium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 215.1 Titrimetric, EDTA, EPA Method 215.2
Chloride	Titrimetric, Mercuric Nitrate, EPA Method 325.3 Colorimetric, Automated Fe <sub>3</sub> (CN) <sub>6</sub> , AAII, EPA Method 325.2
Chlorophyll-a	Spectrophotometric, SM1002G

<sup>\*</sup>See Index to Abbreviations (last page)

<u>Par</u>	rameter	Methodology*
Chr	omium	Direct Aspiration, ICP Furnace, AA, EPA Method 218.2
Cob	palt	Direct Aspiration, ICP Furnace, AA, EPA 219.2
Col	or	Colorimetric, Platinum Cobalt, EPA 110.2 Spectrophotometric, EPA 110.3
Cop	per	Direct Aspiration, ICP Furnace, AA, EPA 220.2
Cya	nide:	
a)	Photometric Determination of Simple Cyanide	Spectrophotometric, EPA 335.1
b)	Total Cyanide after Distillation	Spectrophotometric, EPA 335.2
Eth	yl Benzene	Gas Chromatograph EPA 602, 8020
Flu	oride	Potentiometric, Ion Selective Electrode, EPA 340.2
Har	dness	Colorimetric, Automated EDTA, EPA 130.1 Titrimetric, EDTA, 130.2
Iro	n	Direct Aspiration, ICAP Furnace, AA, EPA 236.2
Lea	d	Direct Aspiration, ICAP Furnace, AA, EPA 239.2
Magi	nesium	Direct Aspiration, ICAP Direct Aspiration, AA, EPA 242.1
Mang	ganese	Direct Aspiration, ICAP Furnace, AA, EPA 243.2
Mer	cury	Cold Vapor, Manual, EPA 245.1

ICAP
46.2
ICAP 49.2
imetric, Distillation 350.2
imetric, Potentiometric,
nated Cadmium Reduction,
onia (see above)
atory Funnel Extraction,
EPA 405.1
lid, High Level, EPA 410.13
Chromatograph 8015
150.1
Manual 4 - AAP, EPA 420.1
Manual 4 - AAP, EPA 420.1
etric, Automated Ascorbic
bic Acid, Single Reagent,

Parameter	Methodology*
Potassium	Flame Photometric Method, SM 322B AA, Direct Aspiration, EPA 258.1
Selenium	AA, Furnace, EPA Method 270.2 AA, Hydride, EPA Method 270.3
Silicon as SiO <sub>2</sub>	Direct Aspiration, ICP Colorimetric, EPA 370.1
Silver	Direct Aspiration, ICP AA, Furnace, EPA 272.2
Sodium	Flame Photometric Method, SM 325B AA, Direct Aspiration, EPA 273.1
Residue: Filtrable (TDS)	Gravimetric at 180°C, EPA 160.1
Suspended	Gravimetric at 103-105°C, EPA 160.2
Settleable	Volumetric, Imhoff Cone, EPA 160.5
Volatile	Gravimetric, Ignition at 550°, EPA 160.4
Conductivity	Specific Conductance, EPA 120.1
Sulfate	Gravimetric, EPA 375.3 Colorimetric, Automated Methyl Thymol Blue, EPA 375.2
Sulfide	Titrimetric Iodine, EPA 376.1
Thallium	AA, Furnace, EPA 279.2
Toluene	Gas Chromatograph EPA 602,8020
Turbidity	Nephelometric, EPA 180.1
Vanadium	Direct Aspiration, ICP AA, Furnace, EPA 286.1
Volatile Organics (Regulated for Drinking	Method 502.2 GC/Hall Detector Water) with Purge and Trap
Xylenes	Gas Chromatograph EPA 602, 8020
Zinc	Direct Aspiration, ICP AA, Furnace, EPA 289.2

Parameter	Methodology*
Digestion Technique for Total Recoverable Metals	Metal S-6, EPA 4.14
Moisture	ASTM D3173
Ash	ASTM D3174
Total Sulfur	ASTM D3177
Pyritic Sulfur	ASTM D2492
Sulfate Sulfur	ASTM D2492
Heat of Combustion	•
Major & Minor Elements	
Trace Elements	ASTM D3683
рН	ASA 60-3
Electrical Conductivity	
Sodium Absorption Ratio	USDA, Page 72
Calcium	USDA 3a, then ICP
Magnesium	USDA 3a, then ICP
Sodium	USDA 3a, then ICP
Saturation	USDA 27
Particle Size	USDA 41
Texture	USDA 41
Carbonate	ASA 91-4

#### ANALYTICAL METHODOLOGIES

<u>Parameter</u>	Methodology*
Selenium	ASA 80-2
Boron	ASA 74-3, 74-4
Nitrate	ASA 84-5
Organic Matter	ASA 92-3
Molybdenum	ASA 74-1
Copper	DPTA, ICP
Lead	DPTA, ICP
Arsenic	EPA 206.3
Potassium	ASA 71-2, 71-4, USDA 11a
Phosphorus	ASA 73-4
Ammonia	ASA 88-3
Exchangeable Cations	USDA 18, 19
Asbestos Content	EPA Interim Method 600/M4-82-020
Asbestos Fibers	NIOSH P&CAM 239

### Abbreviations used in above listings:

SM:

ICP:

EPA: Methods for Chemical Analysis of Water and Wastes EPA 600/4-79-020

<u>Standard Methods for the Examination of Water and Wastewater</u> 16th Edition, APHA-AWWA-WPCF, 1983

Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 200.7, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati,

Ohio, 45268, November 1980.

ASTM: American Society of Testing Materials USDA:

USDA Handbook 60, <u>Diagnosis and Improvement of Saline and Alkali Soils</u>

ASA:

American Society of Agronomy Monograph 9, <u>Methods of Soil Analysis</u>

NIOSH:

NIOSH "Manual of Analytical Methods"

#### 12.0 SIGNIFICANT NUMBERS

The primary objective is to report all results in such a way that they can be interpreted properly with reference to the accuracy of the test.

The attached significant place sheet specifically lists the parameters and the number of significant places that a result should be reported for a specific range. Also included is a minimum detection limit for that parameter. It should be noted, however, that this detection limit is the <u>routine</u> minimum detection limit and often a lower detection limit can be reached by altering the test in some way.

To use the significant place sheet:

- 1. Look up the parameter
- 2. Check to see that the result is not below the minimal detection limits; if it is, the result should be reported as less than this limit.
- 3. Find the range that the result lies within.
- 4. Report the result to the number of significant places as indicated by X's.

Rounding Off Number: The following rules should be used for rounding off numbers to the correct significant places:

364 - 360
487 - 490
365 - 360 375 - 380

TABLE 12-1

REPORT OF RESULTS
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum D Limi		Range	Significant Figures
Asbestos Fibers	Sample Depen		0.001-0.010 0.010-0.100 0.10-1.0 1.0-10	0.00X 0.0XX 0.XX X.X
Asbestos Identification	1	%	1-10 10-100	X
Acidity	1	mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
Alkalinity	1	mg/1	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
Boron	0.1	mg/l	0.1-1.0 1.0-10 10-100	0.X X.X XX
Calcium	1	mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXXO
Calcium Hardness	3	mg/l	3-10 10-100 100-1000 1,000-10,000	X XX XXX XXXO
Chloride	1	mg/1	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
Chlorine Residual	0.1	mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Color	5 col	or units	5-100 100-1000 1,000-10,000	XX XXO XXOO

Parameter	Minimum De Limit	tection	Range	Significant Figures
Cyanide	0.01	mg/l	0.01-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Fluoride	0.10	mg/1	0.1-1.0 1.0-10 10-100 100-1000	0.XX X.X XX XX0
		METALS	5	
Barium (ICP)	0.10	mg/l	0.1-1.0 1.0-10 10-100	0.X X.XX XX.X
Beryllium (ICP)	0.005	mg/1	0.005-0.010 0.01-0.1 0.1-1.0 1.0-10 10-100	0.00X 0.0XX 0.XX X.X XX
Cadmium (ICP)	0.01	mg/1	0.01-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Cadmium (flameless)	0.001	mg/l	0.001-0.010 0.01-0.1 0.1-1.0	0.00X 0.0XX 0.XXX
Cobalt (ICP)	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Cobalt (flameless)	0.001	mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XXX

Parameter	Minimum Detect Limit	cion Range	Significant Figures
Chromium (ICP)	0.02 mç	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.XX XX
Chromium (flameless)	0.002 mg	0.002-0.010 0.010-0.10 0.10-1.0	0.00X 0.0XX 0.XXX
Hexavalent Chromium	0.02 mg	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Grease or Oil	1 mg	1-10 10-100 100-1000 1,000-10,000	X XX XX0 XX00
Hardness, Total	2 mg	/l 2-10 10-100 100-1000 1,000-10,000	X XX XXX XXXO
Iodide	0.01 mg	/1 0.01-0.1 0.1-1.0 1.0-10 10-100	0.0X 0.X X.X XX
Silver (ICP)	0.04 mg	/1 0.04-0.1 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Silver (flameless)	0.001 mg	/1 0.001-0.010 0.010-0.10 0.10-1.0	0.00X 0.0XX 0.XXX
Aluminum (ICP)	0.1 mg	/l 0.1-1 1-10 10-100 100-1000	0.X X XX XX XXO

Parameter	Minimum D Limi		Range	Significant Figures
Arsenic (flameless)	0.001	mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XXX
Barium (ICP)	0.1	mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.XX X.X XX XXO
Copper (ICP)	0.02	mg/1	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.XX XX XX
Iron (ICP)	0.05	mg/l	0.05-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Mercury (flameless)	0.5	ug/l	0.5-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Potassium (flame)	1	mg/l	1-10 10-100 100-1000	X XX XXO
Lithium (ICP)	0.01	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Silica (ICP)	1	mg/l	1-10 10-100 100-1000	X XX XXO
Magnesium (flame)	1	mg/l	1.0-10 10-100 100-1000	X XX XXX

TABLE 12-1 (cont.)

Parameter	Minimum Detection Limit	Range	Significant Figures
Manganese (ICP)	0.02 mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Molybdenum (ICP)	0.05 mg/1	0.05-0.10 0.1-1 1-10 10-100 100-1000	0.0X 0.XX X.XX XX XX
Molybdenum	0.001 mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XX0
Sodium (flame)	1 mg/l	1-10 10-100 100-1000	X XX XXX
Nickel (ICP)	0.02 mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Lead (ICP)	0.1 mg/1	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Lead (flameless)	0.005 mg/1	0.005-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XXX
Antimony (ICP)	0.05 mg/l	0.05-0.10 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.XX XX XX
Selenium (flameless)	0.001 mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XX0

Parameter	Minimum [ Limi		Range	Significant Figures
Tin (ICP)	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10	. 0X . XX X . X
Strontium (ICP)	0.05	mg/l	0.05-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Titanium (IC))	1	mg/l	2-10 10-100 100-1000	X XX XXO
Vanadium (ICP)	0.1	mg/l	0.2-1.0 1.0-10 10-100	. X X . X XX
Zinc (ICP)	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
NITROGEN				
Ammonium (Automated)	0.1	mg/l	0.10-1.0 1.0-10 10-100 100-1000	0.XX X.X XX XXO
Ammonia (Titrimetric)	0.1	mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Nitrates	0.05	mg/l	0.05-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX

TABLE 12-1 (cont.)

Parameter	Minimum Detection Limit	Range	Significant Figures
Nitrites	0.05 mg/l	0.05-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Kjeldahl Nitrogen	0.1 mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
OXYGEN DEMAND			
B0D5	5 mg/1	5-10 10-20 20-100 100-1000 1,000-10,000 10,000-100,000	X XX XX0 XX00 XX000
COD (low level)	0.5 mg/l	0.5-1.0 1.0-10	0.X X.X
COD (high level)	5 mg/l	5-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
Oil or Grease	1 mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
рН	0.1 pH unit	0.1-1.0 1.0-10 10-14	0.X X.X XX.X
Phenol	0.005 mg/1	0.005-0.10 0.10-1.0 1.0-10 10-100 100-1000	. 0XX . XX X. X XX XX0

Parameter	Minimum Lin	Detection nit	Range	Significant Figures
Organic Nitrogen	0.1	mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Total Phosphorus (Automated)	0.01	mg/l	0.01-0.1 0.1-1.0 1.0-10 10-100	0.0X 0.X X.X XX
Ortho Phosphorus	0.01	mg/l	0.01-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX XX
Silica (ICP)	1	mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XXO XXOO
Solids, Total Total, Vol. Suspended Suspended Vol.	1	mg/l	1-10 10-100 100-1000 1,000-10,000 10,000-100,000	X XX XXX XXX0 XXX00
Solids Settleable	0.1	m1/1	0.1-1.0 1.0-10 10-100 100-1000 1,000-10,000	0.X X.X XX XX0 XX00
Specific Conductance	0.1	umho/cm	0.1-1.0 1.0-10 10-100 100-1000 1,000-10,000	0.X X.X XX XXX XXX XXX0
Sulfate	1	mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0

TABLE 12-1 (cont.)

<u>Parameter</u>	Minimum Detection Limit	Range	Significant Figures
Sulfide	0.1* mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XXO
Sulfite	1* mg/l	1-10 10-100 100-1000	X XX XXO
Surfactants	0.05 mg/l	0.05-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX
Turbidity	0.1 NTU	0.1-1.0 1.0-10 10-100 100-1000 1,000-10,000	0.X X.X XX XX0 XX0 XX00
Volatile Organics	Varies; see publish limits	ned 0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX

<sup>\*</sup>Sample volume dependent

#### 13.0 SAMPLE COLLECTION, CONTAINERS AND PRESERVATION

In order to maintain the integrity of the sample from the time it is collected until it is received in the laboratory for analysis, we recommend the use of proper sample containers and preservatives to our clients for the collection of samples. Upon request, we will supply our clients with appropriate sample containers and preservatives. Instructions for sample preservation are given as are material safety data sheets for each preservative supplied.

Certain analyses must be completed in a timely fashion in order to insure that true concentrations are measured. The method of preservation along with the type and volume of preservative used for each parameter is given in Table 13-1. The recommended holding periods are also contained in Table 13-1 for each parameter.

The analyses of those parameters with short holding periods -- 24 hours or less -- are given priority and completed as quickly as possible.

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# TABLE 13-1

#### SAMPLE PRESERVATION

<u>Parameter</u>	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Acidity-Alkalinity	200	Refrigeration at 4°C	Plastic, Glass	14 Days
Benzene*	2 each, 40	HC1 to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Biochemical Oxygen Demand (BOD)	500	Refrigeration at 4°C	Plastic, Glass	48 Hours
Boron	100	Refrigeration at 4°C	Plastic	
Bromide	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Calcium	200	HNO <sub>3</sub> to pH 2	Plastic, Glass	6 Months
Chemical Oxygen Demand (COD)	200	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH 2	Plastic, Glass	28 Days
Chloride	100	None Required	Plastic, Glass	28 Days
Color	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Cyanide	500	NaOH to pH 12 and refrigeration at 4°C 2 ml thiosulfate for chlorinated waters	Plastic, Glass	14 Days

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TABLE 13-1 (cont.)

#### SAMPLE PRESERVATION

Parameter	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Dissolved Oxygen Winkler	300	Fix on Site	Glass	8 Hours
Ethyl Benzene*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Fluoride	500	None Required	Plastic	28 Days
Hardness	200	HNO <sub>3</sub> to pH to >2	Plastic, Glass	6 Months
Iodide	200	Refrigeration at 4°C	Plastic, Glass	24 Hours
MBAS	250	Refrigeration at 4°C	Plastic, Glass	24 Hours
Metals, Total	100	HNO <sub>3</sub> to pH <2	Plastic, Glass	6 Months
Hexavalent Chromium	500	Refrigeration at 4°C	Plastic, Glass	48 Hours
Metals, Dissolved	100	Filtrate: HNO <sub>3</sub> to pH <2	Plastic, Glass	6 Months
Nitrogen, Ammonia	200	H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Nitrogen, Kjeldahl	100	Refrigeration at $4^{\circ}$ C $H_2SO_4$ to pH <2	Plastic, Glass	28 Days
Oil and Grease	1 liter	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Glass	28 Days

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#### TABLE 13-1 (cont.)

#### SAMPLE PRESERVATION

<u>Parameter</u>	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Organic Carbon	100	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Petroleum Hydrocarbons 1) EPA Method 418.1 2) California Method 3) SW-846 #8015	2) 1000 2)	HC1 to pH 2 and refrigerate Refrigeration at 4°C HC1 to pH 2, Refrigeration at 4°C	Glass Glass Glass	28 Days 14 Days 14 Days
pН	50	Determine on Site	Plastic, Glass	2 Hours
Phenolics	1 liter	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Glass, Plastic	28 Days
Phosphorus	100	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Ortho-Phosphates	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Silica	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Specific Conductance	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Solids	500-1000	Refrigeration at 4°C	Plastic, Glass	7 Days
Sulfate	200	Refrigeration at 4°C	Plastic, Glass	28 Days
Sulfide	200	Refrigeration at 4°C 2 ml Zinc Acetate	Glass, Plastic	28 Days
Sulfite	50	Refrigeration at 4°C	Glass, Plastic	48 Hours
Surfactants	500	Refrigeration at 4°C	Plastic, Glass	48 Hours

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TABLE 13-1 (cont.)

#### SAMPLE PRESERVATION

<u>Parameter</u>	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Tannin-Lignin	100	Refrigeration at 4°C	Plastic, Glass	
Threshold Odor	1 liter	Refrigeration at 4°C	Glass	24 Hours
Toluene*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Turbidity	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Volatile Organics in Drinking Water*	2 each, 40	HC1 to pH2 Refrigeration at 4°C	Glass	14 Days
Xylenes*	2 each, 40	HC1 to pH Refrigeration at 4°C	Glass, Teflon Cap	14 Days

<sup>\*</sup>For chlorinated waters, the addition of 0.008% sodium thiosulfate is required.

#### 14.0 EQUIPMENT LIST

Following is a list of equipment currently used by the Chemical and Industrial Hygiene Division. Notes are included as to make, model, and generalized calibration requirements.

Type	<u>Model</u>	<u>Manufacturer</u>	Calibration Frequency
Oven	C-140G	Blue M	Quarterly
Oven	Muffle Furnace	Hythermoco	Quarterly
Oven	***	S/W	Each Use
Oven		Blue M	Each Use
Oven	Moisture Oven	Boeckel	Each Use
Oven	Incubator 0968	Chicago	Each Use
Balance	A-30	Mettler	Daily
Balance	Н6Т	Mettler	Monthly
Balance	( )	OHaus	Monthly
Balance	XAD	Fisher	Monthly
Bomb Colorimeter	1241	Parr	Monthly
Spectro-	•		<b>y</b>
photometer	Spectronic 200	Baush & Lomb	Each Use
Spectro-			
photometer	Turbidimeter	HF	Each Use
Centrifuge		Drucker	None
Shaker		Eberbach	None
Shaker		Eberbach	None
Air Compressor		Sanborn	None
Emission Spectro-			3 / 3 - 3 - 3
photometer	JY48	Jobin Y-Von	Each Use
Atomic Absorption			
Spectrophotometer	951	IL	Each Use
Atomic Absorption			
Spectrophotometer	Video 2200	Thermo Jarrell Ash	Each Use
Microscope	BHT	Olympus	Each Use
Microscope		AŎ	Each Use
Conductivity			
Bridge	310A	Beckman	Each Use
pH Meter	501	Orion	Each Use
Gas Chromatograph	5890	Hewlett Packard	Each Use
Auto Analyzer	SMAC II	Technicon	Each Use
Auto Analyzer	Boron #1	Technicon	Each Use
Auto Analyzer	Ammonia #2	Technicon	Each Use
Auto Analyzer	Phosphorus #3	Technicon	Each Use
Auto Analyzer	Phosphorus #4	Technicon	Each Use
Oven	Drying Oven	SGA	Each Use
Oven	Drying Oven	Despatch	Each Use
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#### 15.0 AUDIT PROGRAMS

Chen-Northern participates in as many performance audit programs as is economically feasible. These audits can take the form of simple round robin analyses or formal audits by regulatory agencies. The following is a list of audit programs in which we are currently enrolled:

- EPA Safe Drinking Water Analysis Certification
- EPA NPDES Performance Audit Program for Wastewater Analysis
- Utah State University Round Robin Soil Analysis
- NBS Asbestos Identification Round Robin for Bulk Material Analysis

NIOSH PAT Program for Fiber Analysis by Phase Contrast Microscopy

EPA Lead in Airborne Particulate for HiVol Filter Analysis

### 16.0 RECIPIENTS OF QUALITY ASSURANCE PLAN

Montana Department of Health & Environmental Sciences Pat Bugosh - Chen-Northern, Inc. - Helena, MT 2. Ed Hart with Montana Power Company - Butte, MT 3. Lab Staff - Chen-Northern, Inc. - Billings, MT Kathie Roos with Special Resource Management 5. Debbie Madison with Fort Peck Tribes - Poplar, MT 6. Morrison-Maierle/CSSA - Bozemam, MT 7. Joe Nosek - Twin City Testing - St. Paul, MN 8. 9. Chen-Northern, Inc. - Denver, CO 10. 11.

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